ORGANIC CHEMISTRY LAB NOTEBOOKS

Learning how to keep a lab notebook is an integral part of an organic chemistry lab. Therefore, one part of your grade will consist of your skill in keeping a lab notebook. Two sample lab notebook entries will be attached. Example 1 (pg. 3) is a technique lab and Example 2 (pg. 8) is a preparation lab.

OVERALL

1. You must have a bound duplicate carbon research notebook. You will be required to turn in the carbon copies at the end of each lab period. The copies must be legible, so be sure to keep this in consideration when taking down information. Nothing should be written directly on the carbon copies – these are copies only.

2. A table of contents is a wonderful idea. It will help you keep track of where everything is.

3. Number the pages of your notebook if they are not numbered already. Do not renumber any pages already numbered.

4. Write on only one-side of the sheets in your lab notebook. I consider the backs to be scratch paper for you and I will not grade them.

5. Use ink. Not pencil!!! No erasable ink either.

6. Don’t erase, scribble-out, whiteout or anything else along this fashion. I know you will make mistakes, this is expected! When you do make a mistake, simply cross out the mistake with a few lines so that the mistake can still be read through the lines. You may even find it useful to put a note to explain why you made the mistake so that you can avoid in the future. However, most mistakes do not need this sort of clarification.

7. Write down everything you do and everything that you observe. Your notebook should be a record of everything that happened in lab. Most important, anyone should be able to repeat the experiment using only your notebook as a guide.

8. Remember to sketch all set-ups in your notebook. Very common techniques, such as vacuum filtration, only need to be sketched the first time you perform the technique.

ADVANCE PREPARATION (PRELAB ASSIGNMENT)

In order for you to get the most out of your laboratory experience, you must prepare before you come to lab. Students who try to do an experiment “cold” – following the written procedure word by word without regard for what is to come – often meet with unexpected challenges that cannot be solved in the allotted time and end by having the experiment fail. You will be required to turn in your prelab notebook entries (part 2, below) BEFORE you start the experiment. In addition, you will be allowed to refer to only your prelab assignment (and not the lab manual) while performing the lab. Therefore, it is essential that your prelab assignment be complete and accurate. The prelab assignment consists of the following:

1. Looking up the properties of each chemical to be used in lab.¹ Some of the information can be found in the experiment itself, and other information may not be relevant.

2. Starting your notebook entry for the experiment. Include the following:
   a. Title
   b. Purpose: A clear, concise statement defining the scientific problem the experiment is designed to solve. Why are you doing the experiment?
   c. Reactions: For a preparation experiment, write the balanced equation for all significant reactions (including side reactions).
   d. Table of Chemical Properties: A table that lists the relevant physical properties or other information (molecular weight, melting point, boiling point, density, potential hazards, source, purity, etc.) about the reactant, products, solvents, and any other chemical involved in the experiment.

¹ An excellent source of such information is the Aldrich chemical catalog, Aldrich Chemical Company, Milwaukee, WI; a somewhat more detailed source is The Merck Index, Merck & Co.: Rathway, NJ. The Aldrich catalog can be found in M 230, on reserve in the Parkland College Library, and is also on the web (http://www.sigmaaldrich.com), where it provides access to a chemical’s MSDS. In addition, information can also be found on the web at Chemfinder.com (http://chemfinder.cambridgesoft.com).
Calculations: For a preparation experiment, calculate the quantity of reactants needed and the theoretical yield of the product.

Prelab Procedure: This should be a detailed outline of how you plan to proceed. Include in this section diagrams of any set-ups you will use, the amounts of chemicals needed, and detailed explanations of how you plan to perform the experiment. The audience for this section is a student of organic chemistry who has never performed the experiment before (that’s you!).

LABORATORY RECORD
When you begin the actual experiment, keep your notebook nearby so you will be able to record those operations that you perform. You should keep a detailed account of your work, reporting everything of importance that you actually did and saw. This section of your notebook should not be prepared in advance. The purpose is not to write a recipe, but rather to record, in your own words, how you carried out the experiment. You should include all relevant data such as the quantities of materials that you actually used (not the amounts in the prelab procedure – your data should differ) any observations that you make (please make special note of time and color, in particular), and the results of any analyses that you performed. Raw data should be recorded with particular care; if you forget to record data at the time you measure it, or if you record it incorrectly or illegibly, the results of an entire experiment may be invalidated. Don’t forget to be as accurate as possible – write down all digits when massing compounds and estimate the last when measuring the volume. When your product has been prepared and purified, or isolated if it is an isolation experiment, record pertinent data such as the melting point or boiling point, its density, and the conditions under which spectra were determined. In addition, you should include a sketch of the apparatus you used if it differs from the sketch given in the prelab procedure. Your notebook should be complete enough that someone else should be able to perform the experiment without using any other source than your laboratory notebook.

You may find it helpful to think of your lab notebook as telling a story about your accomplishments (or misadventures) in the laboratory. Although most professional journal articles are written in a dry, impersonal style, this need not be true of a lab notebook. Feel free to use the first person when writing in your lab notebook. The key features of any good experimental account are neatness, brevity, clarity, completeness, and timeliness. Your audience for this section is an experienced organic chemist (which will hopefully be you by the end of the semester). Therefore, it is not necessary to go into lots of detail about procedures (such as obtaining a melting point, or performing an extraction) that are performed frequently in an organic chemistry lab.

CONCLUSION
After you have finished the experiment, write down your conclusions and explain how you arrived at them. If you are performing a technique your conclusion should always include information related to the technique performance. If you are identifying an unknown, your conclusion should always include the unknown letter, the identity of the compound, and how you identified the unknown (melting point, etc.). If you are preparing a compound, your conclusion should always include the overall and percent yield, the identity of the product, and how you verified that your compound is the expected product (melting point, etc.). Whenever compounds are identified, be sure to include the method of identification, your data, and the corresponding literature data.

REFERENCES
The Separation of a Liquid Mixture
By Distillation

Purpose: To separate two liquids and to practise the technique of distillation.

Table of Chemical Compounds

<table>
<thead>
<tr>
<th>Compound</th>
<th>Molecular Formula</th>
<th>MW</th>
<th>b.p.</th>
<th>density</th>
<th>hazards</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-propanol</td>
<td>C₅H₁₂O</td>
<td>74.14</td>
<td>97.4°C</td>
<td>0.785 g/mL</td>
<td>flammable, irritant</td>
</tr>
<tr>
<td>Water</td>
<td>H₂O</td>
<td>18.02</td>
<td>100°C</td>
<td>1 g/mL</td>
<td>none</td>
</tr>
</tbody>
</table>

Prelab Calculations - include any calculations needed before lab here.

Procedure - be detailed. You will not be able to use your lab manual in lab.

1) Set up apparatus:
- Thermometer
- Thermometer adapter
- Distillation head
- 50 mL R.B. flask
- Condenser
- Heating mantle (Thermowell)

2) Obtain 25 mL of 2-propanol-H₂O mixture in R.B. flask.

3) Add boiling stones to R.B. flask (2-3 stones)
Janet Song
Distillation

2/3/03

4.) Place R.B. flask on distillation head. Get instructor to check apparatus.
5.) Use variac to heat R.B. flask (Variac setting ~ 40 volts)

Mistakes should still be legible! No white out, etc.

6.) Collect distillate in graduated cylinder.
7.) Record temperature for every 1 mL collected.
8.) After 5 mL collected, transfer distillate to test tube. (Label test tube fraction #1)
9.) Keep collecting, noting temp every 1 mL.
10.) After next 5 mL collected, transfer distillate to test tube (Fraction #2)
11.) Continue as above, recording every 1mL (temp) & transferring every 5 mL portion to a new test tube & labeling a fraction.
12.) After 20 total mL total has been collected, remove heat & let system cool.
13.) Label transfer all liquid left in R.B. flask to test tube & label "post:"
14.) Turn in test tube to be analyzed.
15.) Clean up!

Cross off unused portion of page.

This completes the prelab portion for this example. Turn in before lab.
This portion of the notebook can NEVER be written prior to lab.

Janet Song
Distillation

Lab Record - written as you perform the lab. Must consist of what was actually done in lab. Don't forget to include observations.

Laboratory Record:
Set-up apparatus as directed for simple distillation (pict. on pg. 17). Obtained 25.3 mL of 1:1 2-propanol-H₂O mixture. Turned H₂O flow on (thru condenser). Started heating (varia @ 43 volts) @ 3:00 pm. At 3:15 pm, started seeing bubbles. Full boil occurred at 3:32 pm. At 3:45 pm, drops going thru condenser. Temperature is now 78°C.

<table>
<thead>
<tr>
<th>Volume</th>
<th>Temp (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 mL</td>
<td>75</td>
</tr>
<tr>
<td>2 mL</td>
<td>79</td>
</tr>
<tr>
<td>3 mL</td>
<td>81</td>
</tr>
<tr>
<td>4 mL</td>
<td>83</td>
</tr>
<tr>
<td>5 mL</td>
<td>85</td>
</tr>
<tr>
<td>6 mL</td>
<td>85</td>
</tr>
<tr>
<td>7 mL</td>
<td>87</td>
</tr>
<tr>
<td>8 mL</td>
<td>89</td>
</tr>
<tr>
<td>9 mL</td>
<td>89</td>
</tr>
<tr>
<td>10 mL</td>
<td>90</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Volume</th>
<th>Temp (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>11 mL</td>
<td>90</td>
</tr>
<tr>
<td>12 mL</td>
<td>91</td>
</tr>
<tr>
<td>13 mL</td>
<td>92</td>
</tr>
<tr>
<td>14 mL</td>
<td>93</td>
</tr>
<tr>
<td>15 mL</td>
<td>95</td>
</tr>
<tr>
<td>16 mL</td>
<td>96</td>
</tr>
<tr>
<td>17 mL</td>
<td>97</td>
</tr>
<tr>
<td>18 mL</td>
<td>98</td>
</tr>
<tr>
<td>19 mL</td>
<td>99</td>
</tr>
<tr>
<td>20 mL</td>
<td>100</td>
</tr>
</tbody>
</table>

After each 5 mL collected, transferred to test tube and labeled as a fraction.
Janet Song

Distillation

After 20 mL collected (4:30 pm), removed heat and let the system cool. Remaining liquid transferred to test tube and labeled “pot”. Submitted sample for analysis.

End of lab record for this example. Turn in before you leave lab.
This is the remainder of the lab notebook. It should be turned in with the lab report. Your notebook is not complete without it - don't forget to turn it in!

Janet Song
Distillation

Calculations - include all needed calculations for results here. Show work!

Results

<table>
<thead>
<tr>
<th>Fraction</th>
<th>% 2-propanol</th>
<th>% H2O</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>90</td>
<td>10</td>
</tr>
<tr>
<td>2</td>
<td>65</td>
<td>35</td>
</tr>
<tr>
<td>3</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>4</td>
<td>20</td>
<td>80</td>
</tr>
<tr>
<td>pot</td>
<td></td>
<td>100</td>
</tr>
</tbody>
</table>

Results - summary of results obtained during lab.

Discussion

Only pot is pure H2O. All other fractions are mixtures. However, you would expect #3 to be 50:50, so that is reasonable (12.5 mL of each compound in 1:1 mixture).

Discussion - Long explanations go here. This section is especially important to explain your reasoning and non-optimal results.

Conclusion

Simple distillation could separate water from 2-propanol, but did not yield any pure 2-propanol. The purest 2-propanol fraction was 90% 2-propanol; the pot was 100% water. In order to obtain pure 2-propanol, a method other than simple distillation must be used.

Conclusion - Reiterate results (nothing new here). Include brief explanation of results.
The synthesis of 1-bromobutane

**Purpose:** To prepare 1-bromobutane from 1-butanol.

**Reaction:** Reaction you are performing.

\[
\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} + \text{NaBr} \xrightarrow{\text{H}_2\text{SO}_4} \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Br} + \text{Na}_2\text{SO}_4 + \text{H}_2\text{O}
\]

**Side Reactions:** Only important ones.

\[
\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} \xrightarrow{\text{H}_2\text{SO}_4} \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Br} + \text{H}_2\text{O}
\]

Include reagents, products (all, including those from side rxns), and solvents in table.

<table>
<thead>
<tr>
<th>Formula</th>
<th>F.W.</th>
<th>m.p.</th>
<th>b.p.</th>
<th>Density</th>
<th>Hazards</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-Butanol</td>
<td>C_4H_10O</td>
<td>74.12</td>
<td>-89.8</td>
<td>13.5</td>
<td>0.990</td>
</tr>
<tr>
<td>Sulfuric Acid</td>
<td>H_2SO_4</td>
<td>98.08</td>
<td></td>
<td></td>
<td>1.8211</td>
</tr>
<tr>
<td>Sodium Bromide</td>
<td>NaBr</td>
<td>102.9</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1-Bromobutane</td>
<td>C_4H_9Br</td>
<td>137.03</td>
<td>-112B</td>
<td>100.3</td>
<td>1.3764</td>
</tr>
<tr>
<td>Diisobutyl ether</td>
<td>C_{9}H_{18}O</td>
<td>133.25</td>
<td>142</td>
<td></td>
<td></td>
</tr>
<tr>
<td>trans-2-butene</td>
<td>C_4H_8</td>
<td>56.10</td>
<td>2.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>cis-2-butene</td>
<td>C_4H_8</td>
<td>56.10</td>
<td>5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1-butene</td>
<td>C_4H_8</td>
<td>56.10</td>
<td>-5</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Only give important data - no need to include mp, if never solid, for example.
Quinten Quant
Synthesis of $\text{Br}_2$

Always good to know how much product to expect.

Calculations: (theoretical yield of 1-Bromobutane,
using 17 mL of $\text{CH}_3\text{OH}$)

\[
(17 \text{ mL } \text{CH}_3\text{OH}) \left( \frac{0.8908 \text{ g}}{1 \text{ mL}} \right) \left( \frac{1 \text{ mol } \text{CH}_3\text{OH}}{74.12 \text{ g } \text{CH}_3\text{OH}} \right) = 0.1857 \text{ mol } \text{CH}_3\text{OH}
\]

\[
(0.1857 \text{ mol } \text{CH}_3\text{OH}) \left( \frac{1 \text{ mol } \text{Br}_2}{1 \text{ mol } \text{CH}_3\text{OH}} \right) \left( \frac{187.08 \text{ g } \text{Br}_2}{1 \text{ mol } \text{Br}_2} \right) = 35.41 \text{ g } \text{Br}_2
\]

Prelab Procedure:
1. Place 24.0 g NaBr, 25 mL H$_2$O and 17 mL 1-butanol
   in a 250 mL Erlenmeyer flask and cool in an ice
   bath until temp. < 0°C
2. Slowly add concentrated H$_2$SO$_4$ with
   swirling
3. Set up apparatus as follows:

   Apparatus pictures extremely useful.

4. Reflux for 30 minutes (1 drip/second)
Quinolin Quant
Synthesis of Br

5.) Let mixture cool.
6.) Distill mixture. Cool receiver in an ice/H2O bath. Keep distilling until distillate is NOT cloudy.
7.) When distillate becomes clear, collect a few drops in a test tube. Add water and shake. If 2 layers form, distill another 5-10 minutes and repeat this test. If two layers do NOT form, distill another 5-10 minutes and then quit.
8.) Wash distillate with 25 mL H2O.
9.) Wash distillate with 15 mL cold conc. H2SO4
10.) Wash distillate with 15 mL 10% NaOH soln.
11.) Dry with anhydrous H2SO4 and gravity filter into small, dry, RPB flask.
12.) Distill dried product using dry distillation apparatus.

Cross off unused area of page.

End of prelab portion. Turn in at beginning of lab.
Quinoline Synthesis

Laboratory Record:
Placed 0.40g NaBr, 25 mL H₂O and 17.0 mL 1-butanol
in a 250 mL Erlenmeyer flask and let cool in an ice-water
bath. When the solution reached 5°C, added 20 mL
conc. H₂SO₄ with swirling. The mixture warmed up
and turned yellow. Set-up for reflux with gas
trap as on pg. 36. Started refluxing at 10:00 am.
While refluxing, placed 15 mL H₂SO₄ in
Erlenmeyer in ice-water bath to cool for later.
At 10:30 am, let reaction mixture cool to
room temperature, then put in ice water
bath. There were 2 distinct layers in the flask.
Both are orange. Color due to free bromine?
One of the layers is my product.

Distilled the mixture, and collected
everything that came over up to 100°C. (start: 11:00)

Initially, a cloud, white liquid came our (H₂O
+ organic product?) then clear liquid. Stopped
heating, distilling flask, removed receiver and
replaced it with test-tube in beaker with ice
+ water. Heated to distill over a few drops of
a liquid. Added equal amount of H₂O. shook
tube. NO LAYERS FORMED! Replaced receiving
flask and distilled for 5 more minutes.
Quantum Quant
Synthesis of __

Pour 25 mL distilled into a 25 mL separatory funnel, and added 25 mL water. H$_2$O went in upper layer. UPPER LAYER IS AQUEOUS! Lower layer is organic → keep layer layer!

Washed organic layer with 15 mL cold conc. H$_2$SO$_4$ Solution warmed on funnel wash shaker.

Washed organic layer with 15 mL 10% NaOH soln. Added 5 mL H$_2$O → went into upper layer. Teased aqueous layer with litmus paper (red) and it turned blue. Therefore, organic layer is not acidic, either.

Put organic layer into 50 mL erlenmeyer and added anhydrous MgSO$_4$ in small amounts. Cloudy product turned clear and xs drying agent was swirling in the flask. Corked flask and put it away.

End of portion completed in the first day of lab. Turn in before leaving lab.
Quinlen Quaint
Synthesis of ♦

4/18/08

Second week of lab - new date.

Set up for distillation. Removed thermometer + thermometer adapter and put long stem funnel into flask. Gravity filtered product directly into distilling flask. Propped in boiling stand, replaced thermometer and adapter.

Distilled liquid. Collected material that came over from 100 - 103 °C. Start: 9:30 am

1st drop: 10:05 am Stop: 10:20 am.

weight of vial + product: 30.60 g
labeled vial: - 20.25 g

product: 10.40 g

Put product into clean, dry, labeled vial.

End of portion that must be turned in before leaving lab.

Results

yield: 16.40 g
IR: no peak @ 3200 cm⁻¹ (no alcohol)

List all major IR peaks here.

Calculations

% yield: \[
\frac{16.40 \text{ g}}{25.44 \text{ g}} = 64.46 \%
\]

Results and Calculations may be done after lab is ended.
Discussion - long explanation and explanation of less than optimal results. Include improvements for next time.

Conclusion:
1-Bromotetane (b.p. 106.3°C) was formed. 16.40 g (64.46% yield) of product was formed. Product was confirmed by b.p. (100-103°C in distillation) and IR (νC=O at 1699 cm⁻¹).

Conclusion - summary of results (nothing new). Include: name of product, amount, % yield, confirmation of product (with literature or expected values). Also, explain results briefly.

End of lab notebook. Turn in with lab report.