APPENDIX 2

SAMPLE LAB REPORT FOR CHEM 2440

RAW DATA

You should obtain one exercise book for recording raw data. Ideally, this exercise book should be one which allows you to easily remove pages. The raw data for each lab should be written (IN INK) in this exercise book during the laboratory period. **Write on only one side of the pages.** Since the raw data is marked as part of the report, it is important to write legibly when recording the raw data.

The raw data consists of a record of the procedure, observations and data.

The **procedure/observations** section should be a report of what was actually done and observed during the lab period. It shouldn't be simply copied word for word from the lab manual, rather it should tell what was done (and observed) by the experimenter. If you make any mistakes during the lab period or if unexpected results occur, it should be included in this section (this will come in handy when you are attempting to decide on sources of error for a particular experiment). It should be written in the past tense (using the third person passive voice) and avoid the usage of personal pronouns such as "I" or "we". All relevant observations should be included (initial observations of all reagents, observations when mixed, color changes, precipitate formation, etc.). This must be written in your note book as each step is done (during the lab period, not afterwards). The laboratory instructors will periodically perform spot checks to ensure that this is being done properly.

The **data** section should include all data recorded during the laboratory period. This will include masses/volumes used, mass of products obtained, actual weighings, boiling ranges, melting ranges, etc.

All of the above must be done before you leave the lab. **Your demonstrator may require that you get the raw data checked and signed before you leave the lab.**

Before leaving the lab, these pages (containing the raw data) should be removed from your exercise book and submitted for marking. Since it is the actual raw data which is marked, you need
not copy the raw data into the formal report. By writing all of this during the lab period, it will result in less work being required for the formal report outside of lab time.

The raw data will normally be submitted at the end of the period in which the work is done and will be returned to you so that you can finish writing the formal report.

When the raw data is returned to you, you may need to record some additional data such as final masses, melting points, etc. This data should be recorded in the relevant raw data section (preferably, it should be located immediately following the marked raw data) and should be clearly labeled.

FORMAL REPORT

After the raw data has been returned to you, you can then prepare the formal report which is usually due one week after your raw data has been returned. The formal report should be written on “loose” sheets of paper and must be accompanied by the marked raw data.

The formal report should include the following:

TITLE DATE

PURPOSE:

In your own words, explain the purpose of the experiment. State what will be accomplished and briefly explain how this will be done. Mention what procedures will be used and why, but do not go into great detail concerning the procedure (do not include details such as volumes or masses used, etc.). Include balanced chemical equations where applicable. BE BRIEF. Do not simply copy the Introduction from the lab manual.

CALCULATIONS: (if applicable)

Theoretical yield calculation
Percent yield calculation
DISCUSSION:

Comment on the results of the experiment. Did the experiment proceed as expected? If not, offer an explanation for any anomalous results. Comment on the yield (if applicable). If the product has been isolated from a natural source, find the structure of the compound and the expected percent recovery (The MERCK INDEX may be useful for this). If the yield is less than expected, indicate sources of error to explain why this is so. Include any other comments which you feel are relevant.

QUESTIONS:

Answer all questions given in the lab manual.

When the report is submitted, it should have the following format:

The first page should be a cover page which includes the experiment number, Title, your name and your bench number. Immediately following this, the formal report pages should be attached. The raw data pages should then be attached to the back of the lab report (don’t forget to include any additional relevant raw data which has been recently recorded).

If more than one report is submitted at the same time, each experiment should have it's own report.
The following is a sample lab report using an experiment that has (in the past) been used for Chemistry 1031. The reports for Chemistry 2440 should follow this format.

EXPERIMENT 1

DEGRADATION OF ETHYL BENZOATE BY BASE HYDROLYSIS

INTRODUCTION

Every year hundreds of new organic compounds are synthesized or isolated from natural sources (e.g. extracted from plants or fungi). Most of these compounds are very complex. In order to find out the structure, the compounds are broken down (degraded) into smaller parts that have known or easily determined structures.

Ethyl benzoate is an ester of benzoic acid (a carboxylic acid) and ethanol (an alcohol). In this experiment, you will treat ethyl benzoate with dilute aqueous sodium hydroxide. The hydroxide ion reacts with the water-insoluble ester to form a homogeneous solution of sodium benzoate and ethanol. The ethanol is isolated (wet with water!) by simple distillation from the sodium benzoate solution and discarded. The free benzoic acid is released from the remaining solution by treatment with hydrochloric acid, and isolated by suction filtration.

Before you start this experiment, clean the glassware and lightly grease all of the ground glass joints. Use a small amount of the lubricant supplied (on the upper bench top) to grease the joints, then rotate together to form a smooth seal. If excess grease is used it may be wiped off with a towel. When assembling apparatus, use care in clamping to prevent joints from being pulled apart allowing vapours to escape. Do not clamp too tightly or the glass may break. Do not store glassware with the joints connected as they may "freeze" together.
PROCEDURE

Place ethyl benzoate (7.0 mL, density = 1.047 g·mol⁻¹) in a 100 mL round-bottomed flask and add 2.0 mol·L⁻¹ sodium hydroxide (40 mL) followed by 3 or 4 boiling stones. Attach the condenser to the flask in the reflux position. Have your set-up checked by a demonstrator before continuing. Heat the flask gently over a low flame so that the liquid refluxes. The mixture in the flask should be shaken almost continuously to speed up the hydrolysis reaction. When all of the oily drops of the ester have disappeared (about 15 minutes) the solution should be almost clear. Cool the reaction mixture to room temperature by holding an ice bath up around the flask.

When the apparatus is cool enough to handle, remove the condenser; add two or three more boiling stones to the flask; and set up the apparatus for distillation. Forcing glass tubing or a thermometer through a thermometer adapter is probably the most dangerous manipulation in the organic laboratory. Protecting your hands with a towel is advisable if the adapter does not slide easily. Lubricate the glass with glycerol and then ease the tubing or the thermometer through the hole by gripping it near the adapter and twist or push it through in a series of short strokes. The thermometer bulb must be placed in the correct position so that the recorded temperature is the temperature of the vapour and gives the boiling point of the liquid condenses. Heat the flask gently and liquid will soon begin to distil over into the 50 mL round bottom flask. Read the thermometer and record the temperature at which the first drops of liquid begin to collect in the receiver. Continue to heat the flask just enough to keep the distillation going so that about 1 drop of distillate is produced per second. As soon as the temperature changes (5° rise or fall) or distillation ceases, record the maximum temperature reached while liquid was being collected. (in any case, stop the distillation if the temperature reaches 95°C to avoid loss of water from the solution.) Extinguish the burner and allow the apparatus to cool. Measure, and record, the boiling range and the volume of the distillate collected in your data table. The distillate will not be used in this experiment so it should be discarded in the organic dump in the fume hood.

Pour the contents of the 100 mL distillation flask into a 400 mL beaker and place it in an ice bath. In the fume hood, use a graduated cylinder to measure 10 mL of concentrated hydrochloric acid and add it to the contents of the 400 mL beaker while stirring the solution with a glass rod. Allow the mixture to cool to room temperature and collect the precipitated solid using a Buchner flask and funnel. Rinse the crystals with a little ice cold water and then allow the crystals to dry under suction for a few minutes. Let the crystals dry for a week on a watch glass in your locker. Record the mass of your dry product.
TLC ANALYSIS

Obtain two test tubes. Place a small sample of your benzoic acid in one test tube and place a sample of the ethyl benzoate in the second test tube. Label each test tube. To each tube add about 1 mL of acetone (good grade) to dissolve the samples. Spot each of these onto a TLC plate in the same manner as in the TLC lab, using the small capillaries (spot each sample once or twice). Run this plate in a jar containing 20% ethyl ethanoate / 80 % hexane and then observe the plate using the UV lamp.

CALCULATIONS

1. Write the equations leading to the formation of benzoic acid.
2. Determine the limiting reagent.
3. Calculate the theoretical yield of benzoic acid.
4. Calculate the percentage yield of benzoic acid.

QUESTIONS

a. Explain why there were two layers present initially and why a homogeneous solution was formed when the reaction was complete.

b. Why does shaking the mixture speed up the reaction?
SAMPLE LAB REPORT

The following pages illustrate how the raw data for an experiment should be recorded. This should include the procedure/observations, figures and data

Sept. 4, 1997

Raw data for experiment 1 (Hydrolysis of ethyl benzoate)

Table 1: initial masses/volumes used

<table>
<thead>
<tr>
<th>Volume of ethyl benzoate</th>
<th>7.0 mL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume of 2.0 mol/L NaOH (aq)</td>
<td>40.5 mL</td>
</tr>
<tr>
<td>Volume of 12 mol/L HCl (aq)</td>
<td>10.8 mL</td>
</tr>
</tbody>
</table>

Procedure and observations:

7.0 mL of ethyl benzoate (a clear, colorless liquid) and 40.5 mL of 2.0 molar NaOH\(_{aq}\) (a clear, colorless solution) were placed in a round bottom flask. The resulting mixture (containing two colorless layers) was heated and shaken, while under reflux until the mixture was homogeneous (clear, colorless solution with no layering).

The apparatus for a simple distillation was assembled and the mixture was heated to distil the ethanol. (See table 1) Both the distillate and the residue were clear and colorless. The residue was cooled and poured into a 400 mL beaker. Conc. HCl (10.8 mL) was added to the residue and a thick, white precipitate formed. This was isolated by suction filtration and washed with a few mLs of ice cold water to yield a white, powdery solid. This was stored (on a pre-weighed watchglass) in the locker for a week and the mass was determined.

Samples of the crude product and starting material were dissolved in acetone and analyzed by TLC using 20/80 ethyl acetate/hexane as the elution solvent. The plate was observed using a UV light.
Sept. 4, 1997

Table 2: Results of distillation

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>temperature at start of distillation</td>
<td>80.5°C</td>
</tr>
<tr>
<td>highest temperature reached</td>
<td>92.0°C</td>
</tr>
<tr>
<td>volume of ethanol collected</td>
<td>3.5 mL</td>
</tr>
</tbody>
</table>

Table 3: Product yield

| Mass of watchglass + benzoic acid (sept 11) | 79.13 g   |
| Mass of empty watchglass (sept 4)          | 75.24 g   |
| Mass of benzoic acid                       | 3.89 g    |

TLC analysis

<table>
<thead>
<tr>
<th>Ba</th>
<th>Eb</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ba1</td>
<td></td>
</tr>
<tr>
<td>Ba2</td>
<td></td>
</tr>
<tr>
<td>Eb</td>
<td></td>
</tr>
</tbody>
</table>

Solvent: 20/80 ethyl acetate/hexane
Solvent height: 5.6 cm
Experiment 1

Degradation of Ethyl Benzoate by Base Hydrolysis

Purpose:

The purpose of this experiment is to prepare benzoic acid by the base promoted hydrolysis of ethyl benzoate. The equation for this reaction is:

\[
\begin{align*}
\text{C}_6\text{H}_5\text{CO} \text{OCH}_2\text{CH}_3 + \text{H}_2\text{O} & \xrightarrow{\text{OH}^-} \text{C}_6\text{H}_5\text{COOH} + \text{CH}_3\text{CH}_2\text{OH} \\
\end{align*}
\]

This is actually a two-step reaction. The first step involves a reflux with aqueous NaOH. The NaOH cleaves the ester bond producing sodium benzoate (a water-soluble salt) and ethanol (a water-soluble alcohol).

Equation:

\[
\begin{align*}
\text{C}_6\text{H}_5\text{CO} \text{OCH}_2\text{CH}_3 + \text{NaOH(aq)} & \xrightarrow{\Delta} \text{C}_6\text{H}_5\text{CO} \text{O}^-\text{Na}^{-} \text{(aq)} + \text{CH}_3\text{CH}_2\text{OH} \\
\end{align*}
\]

The resulting solution contains sodium benzoate (a water soluble salt) and ethanol. The ethanol is removed by simple distillation (as an azeotrope with water). The remaining solution of sodium benzoate is acidified to convert the soluble salt into insoluble benzoic acid, which is isolated by suction filtration.

Equation:

\[
\begin{align*}
\text{C}_6\text{H}_5\text{CO} \text{O}^-\text{Na}^{-} \text{(aq)} + \text{HCl(aq)} & \xrightarrow{} \text{C}_6\text{H}_5\text{COOH} \text{(s)} + \text{NaCl(aq)} \\
\end{align*}
\]
Calculations:

Theoretical yield of benzoic acid (based on the overall equation)

Overall equation:

$$
\text{C}_6\text{H}_5\text{COO} + \text{NaOH} (\text{aq}) + \text{HCl} (\text{aq}) \rightarrow \text{C}_6\text{H}_5\text{COOH} + \text{CH}_3\text{CH}_2\text{OH} + \text{NaCl} (\text{aq})
$$

Find moles of ethyl benzoate:

$$\text{Mass}_{\text{ethyl benzoate}} = (7.0 \text{ mL}) \cdot (1.047 \text{ g·mL}^{-1}) = 7.3 \text{ g}$$

$$\text{Moles}_{\text{ethyl benzoate}} = \frac{7.3 \text{ g}}{150.18 \text{ g·mol}^{-1}} = 0.049 \text{ mol}$$

Find moles of NaOH:

$$\text{Moles}_{\text{NaOH}} = (2.0 \text{ mol·L}^{-1}) \cdot (0.0405 \text{ L}) = 0.081 \text{ mol}$$

Moles of HCl = (12.0 mol·L⁻¹) · (0.0108 L) = 0.13 mol

Since the moles of ethyl benzoate is less than each of the moles of NaOH and HCl and since this reaction has 1:1:1 stoichiometry, the limiting reagent is ethyl benzoate. The theoretical yield of benzoic acid is 0.049 mol

Theoretical yield of benzoic acid (in g)

$$= (0.049 \text{ mol}) \cdot (122.12 \text{ g·mol}^{-1}) = 6.0 \text{ g}$$

Percent yield = (3.89 g / 6.0 g) · 100 % = 65 %
Discussion:

In this experiment, benzoic acid was produced by the base-promoted hydrolysis of ethyl benzoate. The yield of benzoic acid was 65%.

Since the yield was less than 100%, some of the product must have been lost.

Sources of error:

If insufficient NaOH was used or if the solution was not heated or shaken sufficiently, the hydrolysis would not have gone to completion. This would result in a low yield of benzoic acid. (This is unlikely because incomplete reaction would have resulted in the presence of two layers.) Addition of too little HCl would result in incomplete precipitation of the benzoic acid, and therefore a low yield. (This is also unlikely since the pH of the solution was measured and the solution was found to be acidic.) During the suction filtration, some product may be lost if the water used for the wash is too warm or if too much water is used. Some product may be lost on the glassware during the various transfers.

The TLC analysis revealed that the ethyl benzoate showed a single spot and is therefore assumed to be pure (or at least there is no evidence of any impurity). The benzoic acid showed two spots and is therefore impure. The \( R_f \) for the small spot (Ba2) is approximately equal to the \( R_f \) for ethyl benzoate, therefore this spot is assumed to be unreacted ethyl benzoate (indicating that the reaction did not go to completion and the product still contains a trace of unreacted starting material). The other spot (Ba1) is the main component in the crude benzoic acid sample (larger spot) and is assumed to represent the benzoic acid in the crude sample. Since the benzoic acid did not travel very far on the plate, the benzoic acid appears to be more polar than ethyl benzoate. This is to be expected, since benzoic acid contains a very polar OH bond, that the ester does not have.
Questions:

a.

Initially, the mixture contains ethyl benzoate and aqueous NaOH solution. The aqueous solution (which is mostly water) is quite polar while the ethyl benzoate is relatively non-polar. The ethyl benzoate cannot dissolve in the highly polar water and therefore two layers are present. After the reaction is complete, the products are sodium benzoate and ethanol. Sodium benzoate is an ionic salt which dissociates in water to form ions, therefore it is soluble in water. Ethanol is a highly polar molecule which can form hydrogen bonds to water molecules, therefore it is water soluble. Since both of these products are soluble in water, no layers are seen.

b.

Since the ethyl benzoate and NaOH solution are not miscible, the reaction between the two can only occur at the interface between the two liquids. Since the contact area (surface area) is fairly small, the reaction will proceed slowly. If the mixture is shaken, droplets of the ethyl benzoate will be dispersed throughout the NaOH solution and the reaction can occur on the surface of each of these droplets. This effectively increases the contact area and the reaction can proceed at a faster rate.