CSU Dominguez Hills

Chemistry 311 – Organic Chemistry I Laboratory

Tuesday, Wednesday, and Thursday sections from 1:00-3:50 pm in NSM C-351

Instructor: Dr. Sean Bonness Office: NSM B-302
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Office Hours: M 9:45 – 11:00 am, W and F 9:45 – 12:00 pm and by appointment

Required Materials

- Fessenden, Fessenden, and Feist, *Organic Laboratory Techniques*, 3rd edition
- Laboratory Notebook with Carbonless Copies (University Bookstore)
- Safety Goggles (Instructor approved)
- Ability to access the internet for **BLACKBOARD** (direct link is http://toro.csudh.edu/webapps/portal/frameset.jsp)
- Optional: Laboratory Coat

Course Description: Techniques of separation, purification, and identification of organic compounds. Introduction to organic synthesis. Three hours of laboratory per week.

Laboratory Notebook: Your notebook is to be with you at all times in the laboratory. This is where you record what you have done and all of the data and observations that you take. The lab notebook is not a lab report. Lab reports are written separately and the carbons from your notebook are attached to the end of the report.

Laboratory Reports: Will be due one week after following the conclusion of an experiment (i.e. for the Tuesday section, Simple Distillation Report is Due the week of 9/10). Each report will be worth 20 pts, for a total of 200 pts from all laboratory experiments (10). Report must be typed. See the **Laboratory Notebook Guidelines** section for writing lab reports below.

Lab Data Sheets and Lab Report Sheets: These sheets found within your laboratory manual tell you what kind(s) of data, calculations, graphs etc. need to be included in your laboratory report for any given experiment.

Exercises: Answers to exercises, found in your manual, are to be turned in with laboratory reports.

Practicum: This is a 2 part laboratory final and is worth 200 pts total. For the first part, you will utilize some of the methods/techniques along with analytical techniques studied this semester for identifying an unknown. This is a closed note, closed book exam. There is no talking with lab colleagues and limited assistance from the instructor and lab technician during this time. For the second part, you will be tested via a paper based exam that will have questions pertaining to the labs (reactions, techniques, methods, apparatus, analytical techniques, etc.) used. You will need a scientific calculator. No cell phones or programmable calculators can be used for both parts of the practicum.

Academic Integrity Statement: A university is a community of learners bonded together by the search for knowledge; the pursuit of personal, social, cultural, physical, and intellectual development; and the desire for the liberating effects of an advanced education. California State University, Dominguez Hills (CSUDH) has a culture--an academic culture--shared with other universities and colleges across the nation. Integral to that culture is a set of values such as academic freedom, dedication to teaching and learning, diversity, civility toward others, and academic integrity. Academic integrity is of central importance in the university community and involves committed allegiance to the values, the principles, and the code of behavior held to be central in that community. Integrity concerns honesty and implies being truthful, fair, and free from lies, fraud and deceit.

Grading Criteria: Letter grade will be based on the following system:

The overall course grade will be based on the following percentage of total points possible.

Grade	Percent
A	90 –100 %
A-	88 – 89 %
B+	86 – 87 %
B	80 – 85 %
B-	78 – 79 %
C+	75 – 77 %
C	70 – 74 %
C-	68 – 69 %
D+	65 – 67 %
D	60 – 64 %
F	0 – 59 %

CHE 311 Organic Chemistry I Laboratory Schedule

Laboratory Topic:	**Tuesday**	**Wednesday**	**Thursday**	**Report Due**
Orientation	8/27/13	8/28/13	8/29/13	
Simple Distillation	9/3/13	9/4/13	9/5/13	
Fractional Distillation & Steam Distillation	9/10/13	9/11/13	9/12/13	Simple Distillation
Extraction	9/17/13	9/18/13	9/19/13	Fractional Distillation & Steam Distillation
Recrystallization	9/24/13	9/25/13	9/26/13	Extraction
Melting Point	10/1/13	10/2/13	10/3/13	
TLC/GLC	10/8/13	10/9/13	10/10/13	Recrystallization
TLC/GLC	10/15/13	10/16/13	10/17/13	
1-Bromobutane	10/22/13	10/23/13	10/24/13	TLC/GLC
2-Chloro-2-methylbutane Alkyl Halide Tests	10/29/13	10/30/13	10/31/13	
Dehydration	11/5/13	11/6/13	11/7/13	1-Bromobutane &
Alkene Classification Tests				2-Chloro-2-methylbutane
GLC on Alkene Product	11/12/13	11/13/13	11/14/13	
Lab Practicum	11/19/13	11/20/13	11/21/13	Dehydration & GLC

LABORATORY NOTEBOOK GUIDELINES

I. The Laboratory Notebook: VITAL INFORMATION

It takes practice and lots of trial and error to keep a good laboratory notebook. In organic chemistry, we expect a lot more from you than your previous chemistry instructors did. Your o-chem lab notebook techniques are the kinds expected in a research laboratory. In order to get high notebook scores, keep three things in mind:

- Aim for perfection.

Perfection in the lab notebook means that you follow the guidelines for prelabs, observations, and post labs exactly, and your work is readable. Guidelines will be distributed in your first day of class. Your instructor may seem incredibly picky about your notebook, but he or she is aiming to train you for professional work.

- If you get points off on any section, fix the problem for the next lab by talking to your instructor. Don't let the problem drag on for half the semester before you take action.

In my CHE 311 lab, students get a grading sheet with each returned lab. The sheet shows how many points were lost on each section of the lab, with comments about what was wrong. Comments might read, "Introduction copied from text" or "Incomplete reference" or "Incorrect significant figures" or "Sample labeled incorrectly". Yet the same students turn up with the same problems over and over again. Please talk to your instructor to clarify points off. Take charge of your lab notebook grade.

- Don't wait until the last minute to do prelabs or write-ups.

An incomplete prelab will and can get you sent out of the lab until you finish, or even sent home for the day. Give yourself time to ask questions or have your work checked at office hours.

Dos and Don'ts

- Write in black or blue ink only.
- Never use White-Out or scribble something out. Put a single line through mistakes.
- Write on the right-hand numbered pages only. Make sure the carbon copy is readable.
- Underline the section heading and put it on the top left side for each section. For example, you should have the word **Introduction** underlined at the top of that section.
- Refer to your laboratory handout for more specific details about what your instructor expects.

Your prelab must be complete before you enter the lab. The prelab contains the following sections:

- **Title**
- **Introduction**
- **Procedure**

II. The Laboratory Notebook: TITLE

The title of each experiment is listed in your syllabus. Write the title across the title box on the first page of the experiment. Your instructor may ask you to jot an abbreviated title at the top of other pages of the experiment.

- Ex. **Title**: The Recrystallization of Impure Acetanilide.
- Abbreviated title for following pages: Recrys.

III. The Laboratory Notebook: DATA TABLE

If you arrive at lab without your data table, you may be asked to leave until it is finished. Information for the tables can be found in The Handbook of Chemistry and Physics or the Merck Index (both are available in the back equipment room of the laboratory, in the library, or at your instructor's office hours). You can also find information in your lecture or lab texts.

Your data table should include all reactants (organic and inorganic) and products (organic only), but not reagents used in the purification process.

Ex. **Data Table**

Name	Structure	MM	Data	Moles	m.p.	b.p.	density
Acetic acid	CH_3COOH	60.1 g/mol	7.0 mL	0.12	---	118 °C	1.06 g/mL
Isopentyl alcohol	$(CH_3)_2CH(CH_2)_2OH$	88.1 g/mol	5.0 mL	0.046	---	132 °C	0.813 g/mL
Sulfuric acid	H_2SO_4	98.1 g/mol	1.0 mL	0.019	---	290 °C	1.84 g/mL
Isopentyl acetate	$CH_3COO(CH_2)_2CH(CH_3)_2$	130.2 g/mol	(6.0 g/6.8 mL)	(0.046)	---	142 °C	0.876 g/mL

*Note: Fill out the Data and Moles sections of the table using the amounts listed in the experiment in grams or mLs (even if they are approximate). You should also calculate the theoretical yield for the product and enter it on the table in parentheses. In these calculations you'll determine the limiting reactant and get a rough estimate of the amount of material the reaction can yield. You will actually use slightly different amounts (with different significant figures) than those recommended in the procedure. Your Calculations section will contain the correct data for your experiment, with the correct significant figures.

IV. The Laboratory Notebook: INTRODUCTION

To write a good introduction, you must figure out the point of the experiment. Students who write their introductions without understanding what they're trying to accomplish end up with unacceptable, weird, or hilarious introductions. If you're not sure about the main idea of the experiment, please ask your instructor.

The most common errors that result in a loss of points are plagiarism, writing too much, and missing the point completely.

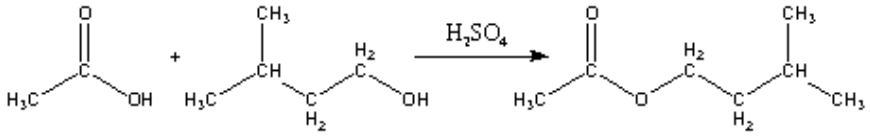
Plagiarism: Don't copy from your handout or text. It is still copying if you change a few words (be especially careful not to include jargon that you don't understand). In the introduction, the ideas must be yours, in your own words. It's okay if your words have a few grammatical errors.

Writing too much: Some students seem to think that the more they throw on a page, the more points they'll get. It just doesn't work that way in organic chemistry. Students end up losing credit because a few scientifically incorrect statements are stuck in the middle of a perfectly good introduction. Don't go on and on trying to impress someone-- just get the job done quickly and efficiently.

Missing the point completely: There is absolutely no reason why this problem should occur. You could take too long to finish the lab, or at worst, you could be a walking safety hazard. Ask your instructor at office hours if you aren't sure what the lab is about. Please don't wait until the lab is about to begin!

For preparation labs, the introduction should describe the main reaction, including the reaction name (if there is one) and mechanism type. Write the chemical equation for the main reaction. If purification or analysis are important parts of the lab, those should be mentioned as well.

- For example, “In experiment 8, isopentyl acetate (banana oil) will be prepared by the esterification of acetic acid and isopentyl alcohol. The ester will be purified by extraction, dried over anhydrous sodium sulfate, and distilled. Product purity will be determined by obtaining and analyzing the infrared spectrum.”



V. The Laboratory Notebook: PROCEDURE

Beginning organic students sometimes have difficulty writing the procedure part of the prelab. The procedure is an abbreviated recipe in your words. It's too hard to follow the paragraph format in the textbook or lab handout. You'll constantly be trying to find your place, and you might miss important steps in the experiment.

- Write out every step, but you don't have to put in every detail.

If the text says, "Assemble a reflux apparatus, using a 25-mL round-bottom flask and a water-cooled condenser. Use a heating mantle to heat. In order to control vapors, place a drying tube packed with calcium chloride on top of the condenser".

The procedure should read, "Assemble a reflux apparatus with flask, condenser, heating mantle, and calcium chloride-packed drying tube."

- For the procedure section of the prelab only, divide the page into two columns. Only write the prelab on the left side of the page. Your in-lab observations will go on the right side of the page.

Procedure	Observations
• **Assemble a reflux apparatus with flask, condenser, heating mantle, and calcium chloride-packed drying tube.** • **Tare a 10-mL graduated cylinder.** • **Add 5 mL of isopentyl alcohol, reweigh.** • **Add 7 mL of glacial acetic acid.** • **Add 1 mL of concentrated acetic acid using a Pasteur pipette.** • **Attach the condenser and drying tube and reflux for 60-75 minutes.**	

- LEAVE PLENTY OF SPACE BETWEEN DIRECTIONS.

Leaving too little space is a very common error. On the observations side of the notebook, you'll put in masses to the correct number of significant figures, details about the appearance of all materials and reaction mixtures, and sometimes explanations about what went wrong and why you had to redo the experiment. If you don't leave extra space for these things, you'll end up squeezing things in or continuing experiments on many different pages. Leave more space than you think you'll need (a complete experiment can take 10-15 pages).

- Make sure your writing is clear and readable.

If your instructor can't read it, she can't grade it. Don't use poor handwriting as an excuse for sloppy work. Your laboratory notebook should be as professional as possible.

VI. The Laboratory Notebook: <u>OBSERVATIONS</u>

Observations are written while you're doing the experiment. It may seem as though you're just rewriting things from the procedures side of the page, but you'll be adding details, the correct significant figures, and explanations.

- Write full sentences.

When an instructor grades observations, he'll only look on the right-hand side of the page. That's why one-word observations like "Done", "3.64 g", or "white solid" are unacceptable.

- Write actions in the past tense. Avoid putting the word "I" in your observations.

Keep it short. An appropriate observation would read something like, "Added 8.94 g of ethyl acetate to the reaction flask. Ethyl acetate is a clear, colorless liquid."
Procedure

Procedure	Observations
• Assemble a reflux apparatus with flask, condenser, heating mantle, and calcium chloride-packed drying tube.	Assembled a reflux apparatus with flask, condenser, heating mantle, and calcium chloride-packed drying tube.
• Tare a 10-mL graduated cylinder.	Tared a 10-mL graduated cylinder. Tare weight = 8.687 g
• Add 5 mL of isopentyl alcohol, reweigh.	Added 5.12 mL of isopentyl alcohol. Isopentyl alcohol is a clear, colorless liquid. Weight of cylinder plus alcohol = 12.438 g Weight of alcohol = 12.438 g - 8.687 g = 3.751 g
• Add 7 mL of glacial acetic acid.	Added 7.24 mL of glacial acetic acid. Glacial acetic acid is a viscous, clear, colorless liquid with a sharp vinegar odor.
• Add 1 mL of concentrated sulfuric acid using a Pasteur pipette.	Added 1.00 mL of concentrated sulfuric acid. Concentrated sulfuric acid is a viscous, clear, colorless liquid. Mixture turned light brown.
• Attach the condenser and drying tube and reflux for 60-75 minutes.	Attached a condensor and drying tube. Heated to reflux. Mixture boiled at 6:50 pm. Reflux was stopped at 7:55 pm (the mixture was refluxed for 65 min). The reaction mixture was brown at the end of the reaction.

- LEAVE PLENTY OF SPACE BETWEEN DIRECTIONS.

Leaving too little space is a very common error. On the observations side of the notebook, you'll put in masses to the correct number of significant figures, details about the appearance of all materials and reaction mixtures, starting times, and sometimes explanations about what went wrong and why you had to redo the experiment. If you don't leave extra space for these things, you'll end up squeezing things in or continuing experiments on many different pages. Leave more space than you think you'll need (a complete experiment can take 10-15 pages).

- Make sure your writing is clear and readable.

If your instructor can't read it, she can't grade it. Don't use poor handwriting as an excuse for sloppy work. Your laboratory notebook should be as professional as possible.

VII. The Laboratory Notebook: CALCULATIONS

Always use the correct number of significant figures in your calculations. Your instructor can help if you're rusty on significant figures.

Most experiments in organic chemistry require only a few calculations. Whenever you make a new product or perform a recrystallization, you'll have to calculate a percent yield.

% yield = (actual yield / theoretical yield) X 100

theoretical yield = (moles of limiting reagent) X (mole ratio) X (molar mass of product)

These calculations are discussed in more detail in your lab text. Other calculations to be performed are described in individual experiments.

VIII. The Laboratory Notebook: CONCLUSIONS

Conclusions should follow directly from your introduction. You should describe how you accomplished everything you set out to do (or explain what went wrong). Discuss the yield, the apprearance, purity, the melting point and/or any spectra. If you made any graphs, you must discuss those. If you're not sure about the main idea of the experiment, please ask your instructor.

The most common errors that result in a loss of points are leaving out important details and stating opinions in the conclusion.

If an example introduction read: "In experiment 8, isopentyl acetate (banana oil) will be prepared by the esterification of acetic acid and isopentyl alcohol. The ester will be purified by extraction, dried over anhydrous sodium sulfate, and distilled. Product purity will be determined by obtaining and analyzing the infrared spectrum."

An appropriate conclusion could read: "Isopentyl acetate was prepared from an esterification reaction of acetic acid and isopentyl alcohol. The crude product was isolated by extraction and drying over sodium sulfate, and then purified by distillation. The boiling range of the product was 80.1 - 84.0 °C. 0.498 g (43.3 % yield) of pure product was obtained. Some of the purified material was lost in the distillation apparatus because drops were clinging to the condenser and because several mL of crude material was left behind in the flask. The pure product was a viscous, pale yellow oil with a fruity odor. The infrared spectrum of the pure product was similar in appearance to the spectrum of isopentyl acetate in the lab text (both major and minor peaks matched in intensity and frequency), except the product IR showed a large water peak at ~

3650 cm^{-1}. Therefore the percent yield may be larger than the actual yield due to the presence of water in the product."

Note the following information that appears in the above conclusion:

- The conclusion tells that the material was prepared, and it mentions the methods of preparation, isolation, and purification.
- The boiling range that was recorded in the observation section is included.
- Both the grams of product and the percent yield are noted. An possible explanation is given for the low yield.
- The product is described. The description must always include the state and color.
- The IR spectrum is compared to the literature spectrum. Specific peaks can also be compared.
- The presence of a water peak in the IR spectrum is mentioned and discussed. Note that water may be introduced during IR sample preparation. This would mean that the pure product is actually dry, but a water peak appears in the spectrum.
- Everything mentioned in the introduction is discussed in the conclusion.

Conclusions tie all the information obtained into one or two readable paragraphs. They follow from the specific scientific results of each individual students. For one lab, I saw two student conclusions that surprised me. One read, "The reaction gave a very low yield, 74.2 %", and the other read, "The reaction gave a high yield, 39.0 %". Low or high compared to what? These are both opinions. A correct statement would contain the percent yield, but not contain any opinions about the number. Words to avoid that often signal an opinion include high, low, very, great, satisfactory, unsatisfactory, well, good, and bad.

A really bad conclusion for the above experiment might read: "The product was made in 20 % yield. The lab went well. An IR was taken. In the isolation procedure, much of the excess acetic acid and the remaining isopentyl alcohol are removed by extraction with sodium bicarbonate and water. The lab was successful."

Note the following problems with this conclusion:

- The name of the product and appearance are not mentioned.
- The mass of obtained product is not mentioned. There is a significant figure problem with the yield, and no explanation of the yield is given.
- Never write "The lab went well." or "The lab was successful." Those are opinions.
- So, an IR was taken. What did it show?
- The "In the isolation procedure..." sentence seems to have a different tone that the rest of the conclusion. That's because it was plagiarized from the textbook.

As always, don't start your conclusion a half-hour before lab. Start it early so you can ask your instructor if anything is missing.

IX. The Laboratory Notebook: <u>GRAPHS</u>

Graphs must be drawn on good quality graph paper (10 squares/cm), or printed using a computer graphing program. Don't draw graphs directly on the lab notebook pages.

The following features should be present (and may be graded) on the graph:

- Put the title of the experiment, a colon, and then the title of the graph at the top of the page. (Hydrolysis of an Alkyl Chloride: Time vs. Log (c/c-x) for Three Solvent Mixtures)
- If more than one set of data will be plotted, put a key in an open space on the graph that shows which symbol refers to which set of data.
- Even if you fit a line or a curve to the data, clearly show all relevant data points.

- Label the x- and y-axes with the appropriate labels, including units. Generally the x-axis should show the independent variable and the y-axis should show the dependent variable. If you're unsure about which is which, ask your instructor.
- Make sure that the scale of the graph is correct. Sometimes students use exponential notation on the scale and the points are not equally spaced.
- The graph should fill most of the page. Don't turn in a graph that is squished onto a quarter of the page.

X. The Laboratory Notebook: <u>Submitting Products</u>

You'll be preparing and submitting compounds throughout the semester. Please don't be lazy. Some common point-busting errors include using masking tape instead of a proper label, submitting the vial without a lid or improperly sealed, writing illegibly, not reporting melting or boiling points as a range, using the incorrect significant figures for temperature or yield, or not following the format specified by the instructor.

Submit solid products in snap-top vials with a label attached. Your lab text gives the proper format for labels:

Name of the compound **Melting or boiling range that you determined** **Yield in grams, and % yield** **Your name**	**Ex.** **Caffeine** **m.p. 74.3 - 78.0 ^{o}C** **0.234 g (10.1 %)** **Michael Rich**

Note that the melting point or boiling point is given as a range, and it should have the correct number of significant figures as determined by the thermometer used. Both the grams in the container and the percent yield are also shown on the label.

Liquid samples are submitted in a snap-top vial that is sealed with parafilm. If the samples are improperly sealed, your product could evaporate away or become contaminated before it is graded. Your instructor will show you the correct way to use parafilm.