Marshall University Department of Chemistry

Chemistry 361 Spring 2008, Dr. R. Morgan

Credit: 3 hours
Co-requisite: CHM 356
Hours Section 201/202 MW 1-1:50 S465, MW 2-3:50 S412/S414
Office Hours: (S-486): MW 11-12, TRF 1-3. (or email me for appt).
Tel: 304-696-3159;
Email: morganr@marshall.edu
Required Text:, Lampman, Kirz and Engel, *Introduction to Organic Laboratory…: A Microscale Approach* (4nd ed.)
Other required items: eye protection, bound laboratory notebook, paper towels.
Recommended: Laboratory coat or apron.

COURSE SCHEDULE

1/14	Introduction, Safety, Calculations, Notebook, Check-In. Read pp 542-580.
1/16	Safety Quiz. Techniques, Instrumentation.
	Exp 1 Introduction to Microscale Laboratory. Lab Exercises 1A, 2.
	Exp 3A Crystallization. T **8-11**. Q (p 32) 1-3.
1/23	Exp 14A Oil of Cloves (part 1). T **5-7**, **12**, **18**, **25**. Q (p 116) 1-3. Read pp 108-116.
1/28	Exp 14A (part 2). Exp 45 Benzocaine (part 10. T 8-11. Q (p 373) 2-5. Read pp 367-373.
1/30	Exp 45 (part 2).
2/4	Exp 28 Nitration of Methyl Benzoate. T 11. Q (p 232) 1-3, 5.
2/6	Exp 25A 4-Methylcyclohexene (part 1). T 5, 6, 12, **13**, **14**, 25. Q (p 216-217) 1-3, 5.
2/11	Exp 25A (part 2). Exp 66 An Oxidation Puzzle (part 1). T 12, 25.
2/13	Exp 66 (part 2). Prepare equipment for Exp 38A.
2/18	Exp 38A Triphenylmethanol (part 1). T 4, 5, 7. Q (p 327) 1, 2, 5.
2/20	Exp 38A (part 2). Begin Exp 36A Preparation of Benzoin by Thiamine Catalysis.
2/25	Exp 35B Reduction of Camphor to Isoborneol. T 6-9, 12. Q (p 301) 1-3.
2/27	**MIDTERM EXAM**. Exp 36A Preparation of Benzoin by Thiamine Catalysis. T 6-9, 11, 12.
	Q (pp 308-309) 1, 4. [Note: same techniques apply to Exp 36B and 36C.]
3/3	Exp 36B Benzil.
3/5	Exp 36C Benzilic Acid. Q (p 314) 1, 2.
3/10	Exp 44C 1,4-Diphenyl-1,3-butadiene. T 8. Q (p 366) 1, 2, 4.
3/12	Exp 42 Preparation of an α,β-Unsaturated Ketone via Michael and Aldol Condensation
	Reactions (part 1). T 7, 8, 11, 12. Q (p 347) 1-4.
3/17	Exp 42 (part 2).
3/19	Lecture I Identification of Organic Compounds (ID of OC). Read pp 448-494, 971-975.
3/31	General Unknown I-III. Begin
4/2 to 4/28	Continue General Unknowns
4/30	Last Report Due. Cleanup. Check-Out.
TBA	**FINAL EXAM (see University schedule)**

There will be no make-up exams or experiments.

Grading Distribution	
Reports	**30%**
Instructor Evaluation	**10%**
Midterm	**30%**
Final	**30%**

The purpose of this course is to:

(1) Introduce you to basic laboratory skills and provide you with experience in interpreting experimental data.

(2) Provide laboratory experience that emphasizes and reinforces the principles and concepts of chemistry in CHM 355 and 356.

(3) See the description of 361 in the online catalog for further information

Safety Precautions

NO Shorts or Sandals! Keep your safety glasses in your drawer so you don't forget them at home!

PASS THE SAFETY QUIZ ON WEB CT BEFORE LAB ON MONDAY!

1. Anyone who has not signed the statement acknowledging one's full understanding of the required safety measures will not be permitted to work in the laboratory.

2. Use care in following the directions of your instructor and laboratory text. Do not alter the experimental procedures without being instructed to do so by the instructor or the TA's.

3. Protective eye goggles must be worn in the laboratory at all times. Failure to do so will constitute sufficient grounds for dismissal from the laboratory. You are responsible for obtaining a pair of safety goggles. **We strongly urge you not to wear contact lenses.**

4. Clothing: Slacks or dresses cut below the knee must be worn. Shoes covering the bridge of the foot and toes must be worn. You will not be allowed to work while violating either of these rules. You cannot wear shorts to lab!

5. Know the locations of all safety equipment in the laboratory. You will be tested on this.

6. All injuries, no matter how trivial, must be reported to the instructor immediately.

Materials Needed

1. Laboratory textbook.

2. A bound laboratory notebook. All experimental data must be recorded directly in this notebook during you laboratory period. You can use either a composition notebook or the scientific notebook sold in the bookstore.

3. Safety goggles; an apron is optional, but desirable.

4. Bring a roll of paper towels to use in lab. MU does NOT provide paper towels (sorry) and you will definitely need some!

Format of the Course

1. Attendance is required in this course.

2. The first half-hour of the pre-lab will be spent in a discussion by the instructor on the experiment to be done in that period.

3. The bound notebook is for the immediate recording of all experimental operations and observations made during the laboratory period. Use only pen to write in the lab books and do not use white-out. If you make a mistake, just draw a line through the mistake and continue writing.

4. Lab reports are due two class periods after the completion date. They should be word processed, with any organic reactions/structures prepared with chemical drawing software. I.e. ISIS draw. They should be stapled and turned in at the beginning of the period.

During exams, talking and sharing of calculators is forbidden. Students will sit in alternate rows while taking the exam.

YOU NEED TO BUY A CALCULATOR THAT DOES ONLY SIMPLE MATH (addition, subtraction, multiplication and division). Calculators that can be programmed or have graphing capabilities are **not** permitted for quizzes. Usually you can purchase one of these cheap solar calculators for $1-$2 at a dollar store.

An Overview of the Key Elements of Your Laboratory Course

Lab Notebook

All data must be recorded in your lab notebook. You should bring your lab notebook to the digital balances to record initial and final weights. Data should NOT be recorded on separate pieces of paper. The format for the lab report will be discussed shortly.

Exams

Fifty percent of your grade is based on in-class assessments (exams). Needless to say, if you do not perform well on exams, obtaining a good grade in this class will be difficult. Neither the midterm nor the final are cumulative. Your average on the midterm and final is the single largest influence on your grade. Take them seriously and you will be successful in the course.

Lab Reports

Copying lab reports: I want to be very clear on this. If you submit a lab report that is very similar to another student's lab report, you will both receive **a grade of zero** for the lab report. End of subject. If you lend your lab report to someone, you are taking a chance that someone will plagiarize your lab report. You could end up with a zero. Several students submitted virtually identical lab reports last semester in CHM 217 and I do NOT want this to happen this semester. Do not copy another person's answers to questions in the lab report. You will both receive zero points if you both turn in identical answers to the questions.

Course Grades

Your grade will be calculated based on your exam grades and your lab report grades. Therefore, it is important to understand the theory behind the experiment, as well as being a good synthetic chemist. The goal of this course is to introduce you to organic lab techniques, as well as to provide you with an opportunity to understand how organic synthesis is performed.

The University scale will be used for grading. I make the average of all student averages completing the course a B (80). Students with 90's receive A's students with 70's C's and so on. Generally the average of student averages are 80 or above. In the event of a class average above 80, I don't scale down. 80 is still a B.

If you have any questions, please do not hesitate to ask me or the teaching assistant(s). I will not embarrass or intimidate you, or at least minimally. Remember that we all make mistakes, but mistakes can be minimized by adequately preparing before lab!Format and Grading of Lab Reports: CHM 361

a) Title

b) Introduction** (20)

a) Purpose

B) balanced chemical equation

c) Mechanism

d) Side reactions, including mechanism if known

e) Data tables w/ references

c) Experimental (20)

d) Calculations (10)

e) Results and Discussion (20)

- You should compare your yield to a known literature yield, if possible using either google.com.

f) Conclusions (5)

g) Exercises or Assigned Questions (15)

h) Compound Purity and Quantity (10)

Lab reports are due the second class period after you have completed the lab. For example, if you completed a lab on Tuesday, you would hand in a lab report on Thursday. Late fees remain the discretion of the grading TA. You can generally expect 10% loss per day.

Comments about Laboratory Notebooks

The purpose of the lab notebook is to provide a place write down all of the relevant information pertaining to the laboratory experiment. Include a title for each experiment and use it to make notes when you read the experiments and listen to the lecture. During the experiment use it as a place to write experimental data, and to make notes. The information is then used to write a laboratory report (see below for details)

Comments for Lab Reports:

1) You should have a table of Physical Property Data. It needs to be done before you go into lab and it should contain reactants and products. You do not need two tables! This table should contain the relevant physical properties of the chemicals you are using, including solvents. Important data includes: molecular weight, m.pt,

b.pt., density for liquids, grams used, moles used. If a liquid was measured using a graduated cylinder only, then you should list volume used.

2) You should have one page just for calculations. On this page put all masses of vials, the limiting reagent calculation and theoretical yield calculation if applicable, etc. The determination of % yield should also be here and make sure it is correct!

Let me be very clear here! You cannot determine whether a reactant is limiting by using your intuition. For example, you need to calculate the moles of each reactant used and then take into account stoichiometry to determine limiting reagent. YOU CANNOT STATE THAT A REACTANT IS LIMITING IF NO CALCULATION IS DONE!

After you calculate the limiting reactant, then determine the moles of product you expect. When calculating % yield, use this formula:

% yield = actual moles obtained/theoretical moles X 100

3) Not all reactions require mechanisms. Ask if you need one in the prelab lecture

4) The experimental section is the most difficult for students to write and to read. LET ME BE CLEAR HERE. I WANT A BRIEF OVERVIEW OF WHAT YOU DID, IN PARAGRAPH FORM, and IN LEGIBLE HANDWRITING. You can combine several steps in one sentence in the procedure. For example:

In a tared 5mL conical vial were placed 4-methyl cyclohexanol (1g, 0.1mol), phosphoric acid (0.4 mL, 0.001mol), and 4 drops of concentrated sulfuric acid. The vial was equipped with a spinning vane, Hickman still and a water-cooled reflux condenser. The reaction was slowly brought to reflux over a 40 minute time period during which 4-methylcyclohexene co-distilled with water. Saturated NaCl was used to rinse out the Hickman still. The layers were separated (note spelling of this word: separated) and the organic layer was dried over sodium sulfate and then stored in a vial. An IR was taken in addition to a b.pt determination using a Meltemp.

In six sentences I told you what I did. Aim for brevity! NOTE THAT THE PASSIVE (THIRD PERSON) TENSE WAS USED. DO NOT USE THE WORDS “I” OR “WE”, etc.

Be as brief as possible. You need to assume that the person reading this knows how to clamp a vial and set up a reaction. You do NOT need to tell me every detail. However, you should specify amounts because they determine yield!

5) It is important to have a stirrer that stirs vigorously. You are often trying to get two immiscible phases to mix and you need a stirrer which really stirs. If you do not have one, search for one in the lab! When you need to stir a reaction, the setting should be on 4-6; the higher, the better, usually. The vial should be in the middle/center of the hot plate.

6) You should compare b.pts and m.pts. to LITERATURE values. There is no such thing as an actual m.pt or theoretical one! There is usually a correlation between purity and physical property data. If a solid has impurities, its m.pt is usually lowered. If a liquid is impure, it depends on the impurities as to whether the b.pt will be elevated or depressed. If the impurity is a solid, the boiling pt will be raised. If the impurity is a liquid and it is more volatile than the synthesized compound, the b.pt will be lowered most likely. The converse is also true.

7) You should always compare your m.pt or b.pt to the LITERATURE VALUE. It is important to decrease the rate of heating when determining the m.pt. when you are within 10C of the literature value. Otherwise your value could be higher than it should be.

8) It is important to use the amounts of each reactant specified in the book even if you are using an automatic pipette. Some students may use less of a reagent than they should because the automatic pipette did not dispense sufficient reagent to the vial. I suspect some students are not using it properly. If the pipette doesn't end up giving you the required mass you need, then dispense more to your vial.

9) In all experiments, but especially in microscale, having enough product is key. There will be some losses due to the compound sticking to the vial, etc. You don't need to mention these because we all know they exist. But the smaller the mass of the compound obtained, the greater will be your losses due to the product sticking to the walls of the vial. If an experiment calls for 1 g, then use 1g. No exceptions. It is better to use slightly more than slightly less!

DO NOT USE HUMAN ERROR AS A REASON FOR LOW YIELD! I know you are human, really I do. There will always be some solid left on the filter paper or some liquid left in the vial. However, it should NOT effect your yield by more than 1 or 2%. Therefore, don't mention it because I realize this error already. However, if you spilled half of your product on the floor, then mention that!

10) If someone tells you that volatility is an issue, then write that one of the possible losses is volatility. This means that the vapor pressure of the product is low and it can be lost simply by sitting in the well of the Hickman still. It also means that you could loose some due to the fact that the water condenser is not as effective as a dry ice condenser. Again, this problem is magnified when there are small masses of products.

11) When doing acid/base extractions, make sure the layers are mixed well or mixing will be inefficient and your product will not be pure.

12) Additional Points

1) You should write a current date in the procedure section when you do an experiment over a two-day period. The procedure should be in paragraph form and concise. Spelling should be correct: SEPARATE

2) If a reagent is formed during a reaction (PhMgBr), then list it in the table of physical properties. List its molar mass and any other information you can find on it that would be relevant.

3) If you write a side reaction that is not discussed in class for a given reaction, it would be nice to know what you are basing this on. Give a reference like the page in your textbook. Dehydration of a primary alcohol to an alkene is a slow process so this isn't a very likely reaction unless it is done in just sulfuric acid. It is possible though. Again, if you determined the exact reaction conditions by doing a search on SciFinder for the dehydration of isoamyl alcohol, then I would be more willing to accept that as a side reaction and I would be very impressed.

4) You need to make sure that your calculations have the right number of significant figures.

5) Realize that recrystallizations do purify products but they are also responsible for yield loss. If you determine a % yield for the crude product and one for the pure product, you could get a good idea of how much material is lost due to recrystallization. Of course, some of that loss is desired because it represents impurities. But do not use excess recrystallization solvent and remember to wash your crystals as sparingly as possible.

6) Results and Discussion Section:

When you obtain a yield for a reaction, it would be good to know how good a yield it is. For example, let's say you obtain a yield of 50% on a reaction. If there are many side reactions, that yield may be a good yield. If, however, the reaction should go to completion, then a 50% yield is poor. So what kind of conclusion can you reach?

Far more impressive is to do a google.com or web search for the experiment you just completed. If you can find the results from someone who performed the reaction under similar conditions, then you should also compare your results to those obtained from the web. Please include a print out from the web.

The Bottom Line

Most people within a very short time will have no problem obtaining 90% on lab reports, or higher. If the first two lab reports have low grades, don't worry about it too much. You will be surprised at how much you improve. Your grade will be determined by your performance on exams more than your lab report grades. Trust me on this one! So if you want to worry about something, worry about the quizzes and not the lab reports.

In the final analysis, what I want to know is to what you comprehend what you are doing in lab. Are you just following a recipe or do you understand the chemistry that you are doing? That is why I emphasize the exams so much. Every instructor of CHM 361 feels the same way, so I am not unique in this respect. Therefore, make sure you understand the pre-lab lecture, the calculations involved and the reason you are doing the experiment.

Overview:

Label each section of your lab report so it is clear what is in each section. Write the procedure as concisely as possible in paragraph form (ask me if you need help here). Keep all of your calculations in the calculation section and make sure all the calculations you are supposed to do are written in that section. Make logical conclusions in the discussion and conclusion section. If you discuss a side reaction, then cite the source for that reaction or a similar reaction.

Good luck!