Chem 216: Synthesis and Characterization of Organic Compounds

Laboratory Syllabus

Chem 216 Section 1xx – Tuesday, 1:10-5:00 pm; Room A000

GSI: name

Email: uniquename@umich.edu

Class participation

- Chem 216 consists of 10 experiments, with students performing all labs, lab reports, and quizzes individually. *The plagiarism policy of UM will be strictly enforced.*

Attendance

- You are expected to attend all classes
- Classes will start on-time at 1:10 pm. You get one "freebee" but If you are consistently late, you will lose GSI points
- If you know you are going to miss a class and have a legitimate excuse, let us know in advance
  - You need to email me (zzzzz@umich.edu) as well as Dr. Koreeda (koreeda@umich.edu). Oral reminders are sometimes forgotten!
  - The further in advance I know, the better we can make-up labs quickly and keep you on track.
- If you are sick/ have an emergency and are going to miss lab, please email me and Dr. Koreeda as soon as possible.

Grading procedures

- Lab reports, quizzes, attendance, and performance in lab will compose two-thirds (67%) of your final grade, and will be assigned by me.
- The other one-third (33%) of your grade will be based upon two exams given by Dr. Koreeda.

Breakdown of Lab grade

Pre-lab reports	5 pts per lab session X 10 expts	50
Lab Reports	25 pts per expt. X 7 expts.	175
Quizzes	33 pts per quiz X 3 quizzes (+ 1 point)	100
GSI evaluation	10 pts per session X 11 sessions	110
Total Points		**435**

- I try to be as consistent as possible when grading lab reports and quizzes within my section. If you notice a discrepancy, please let me know as soon as possible and we will work to rectify it.

Remember, students are evaluated within their own lab section (not between other sections) for final grades. This helps ensure that students with different GSI's are evaluated similarly for similar efforts.

What you can expect from me:

- Availability outside of class
  - Office hours: XXXday, x-y AM in Alcove X of the SLC
  - Email: uniquename@umich.edu
    - I will try to get back to you as soon as I can between the hours of 10 am and 10 pm.
- If you need additional assistance with assignments
  - Other GSIs are available for 215/216 throughout the week. They are a helpful group of people, so if I am not available, please go see them if you have immediate questions. Check the SLC GSI hours schedule online for more information.
- I will try to return all assignments back to you one week after they are given to me.

SAFETY

- Safety goggles are required to be worn at all times. No contacts allowed!
- Please wear closed-toed shoes (i.e. no ballet flats/ sandals/ flip-flops) and appropriate clothing (pants/ long skirts preferred). I will send you home to change if I feel you are dressed improperly.
- Aprons and gloves must be worn when working with chemicals, especially carcinogenic and corrosive ones.
- Gloves should not be worn outside the lab or when handling personal items (i.e. cell phones)
  - No cell phone use is allowed in lab.
- Keep chemicals and reactions in the hood and only in the hood. No reactions should be carried out on the benchtop. Most of the chemicals you will be working with are not fun to breathe.
- No headphones are allowed to be worn in the lab.
- Do not sit on the floor—it is dirty and has who-knows-what chemicals on it. Likewise, be careful about resting bare skin on table-tops; if someone has spilled strong acid/ base, you will feel it.
- Be very aware of flames or things that are hot. Hot plates don't necessarily look hot but you could get burnt. Treat such equipment as if it has just been turned on.
  - Solvents should be moved as far away as possible from heat sources
  - Don't leave hot plates or mantles on when not in use

WASTE AND CLEAN-UP

- All waste (including gloves) should go into the proper waste bucket or bottle
  - Solids: go into the white buckets (little to no solvent should be present)
  - Liquids: go into appropriately labeled bottles in the waste hood (little to no undissolved solid present)
  - NOTHING GOES DOWN THE DRAIN – you don't want to end up drinking it!
- Keep your own areas clean as well as the balances areas—if you make a mess, clean it up—I will deduct GSI points for messes left for me to clean up.

GOOD TLCS

- Use THIN capillary tubes for spotting
- Dissolve all compounds (even liquids) in CH_2Cl_2 (or an appropriate solvent)
  - The more dilute the sample, the better the results (several mgs in 1-2 mL)
- Spot lightly on the plate

    - If you are unsure if anything was transferred, check under UV light before running the plate
- Choose an appropriate solvent system
    - Generally a combination of ethyl acetate and hexanes
    - 50:50 ethyl acetate : hexanes is a good place to start, but try something more/less polar if resolution is poor

GRADING (PRE-LABS, LAB REPORTS, QUIZZES)

Pre-Labs (***50 points overall, 10 at 5 points each***)

- Will be required for all lab sessions starting the second half of Experiment 1 (i.e., Tue, 5/10) with the exception of the second half of Experiment 7 and Experiment 8.
- Graded for completion, though may be randomly collected for more extensive grading
- General Guidelines
    - Appropriate Title
    - Brief Introduction to experiment (concepts, techniques)
    - Simple reaction scheme
    - Table of Reagents (*sometimes not necessary, only use what is necessary for that experiment)
        - Compound Name (or structure)
        - Molecular Weight (g/mol), Density*, Concentration*
        - Amount (g or mL)
        - Number of moles (or mmol)
        - Number of Equivalents*
        - Additional physical constants* (melting point or boiling point)
        - Safety information
            - Is it flammable? Carcinogenetic? Corrosive? Look up compounds using MSDSs [Material Safety Data Sheets].
    - Calculations!
        - Calculate the theoretical yield (how much product should you get?)
        - Molarity/ solution calculations (if making your own)
    - A flow chart of experimental steps (not procedure copied word for word, abbreviated)

Quizzes (*100 points, 3 at 33 points each + 1 pt*)

- 3 announced quizzes throughout semester on material or techniques covered in the lab or talked about lab lecture. More specific information announced the lab before the quiz.

Lab Reports (***175 points; Expts #1, 2, 3, 4, 6, 7, and 8 at 25 points each***)

- Lab reports are due two weeks after the scheduled completion date, to be handed in at the beginning of the lab period.
- Late Reports
    - If submitted a day late: 5 pts off
    - More than a day late: 50% off from the total lab score
    - More than a week late: no points will be awarded

- Reports should be concise (**Not more than 3 pages**, 12 pt font, doubled-spaced or 1.5 spaced, 1" margins).
- The structure of a lab report: Written in present tense
  - Brief introduction (3 points)
    One paragraph long
    Talk generally about the concepts being covered in the lab experiment
    Why is this reaction/ substance important?
    What lab techniques are we using?
    How will we know if we have product?
  - Reaction scheme (2 points)
    Drawn using ChemDraw
    Starting materials, reagents over reaction arrow, products
    This is not a mechanism
    Only include reagents that are necessary to the reaction (no solvents, no workup of the reaction)
  - Experimental procedure (5 points): Written in past tense
    About 1 paragraph long (and written in paragraph form – no bulleted list)
    You don't have to detail every part of the experimental process in your lab reports (i.e. you don't have to tell me a thermometer was used to make temperature readings). Things that would be common knowledge for the experimenter can be left out; after all you didn't describe how you measured out you compounds. Things like extractions are done by chemists everyday, so details like what layer was what and the various flask transfers you made, aren't necessary and just take up space. For special techniques or setups that are seemingly unique to our lab, reference the notes, book, etc.

Here is what I would call a good example of an experimental description found in chemistry literature:

A 100-mL, round-bottomed flask containing a magnetic stir bar equipped with a reflux condenser and argon inlet was charged with 4-bromophenol (3.65 g, 21.1 mmol) and potassium carbonate (5.46 g, 39.5 mmol) were combined. Dry acetone (44 mL) was added resulting in a white, heterogeneous mixture followed by 1-bromobutane (1.4 mL, 1.80 g, 13.2 mmol). The reaction mixture was vigorously stirred and heated to reflux. After 2 d, the mixture was cooled to rt and filtered through a medium porosity fritted funnel. The solids were washed with acetone (30 mL) and the combined filtrate was concentrated in vacuo. The residual oil was dissolved in dichloromethane (40 mL) and the resulting solution washed successively with 3 M aq NaOH (2 x 10 mL), H_2O (30 mL), and brine (30 mL) and dried over anhydrous $MgSO_4$. The organic solvent was removed in vacuo and the resulting oily residue purified by short-path distillation to afford 2.28 g (75 %) of product as a colorless oil. The spectroscopic data matched those from the literature.

Now this is certainly not the only way to write them, and more detail may be appropriate for your reports, but I think it should give you a feel for how one should be written.

- Results and discussion (11 points)
  - Written in 3rd person past tense
  - 1-2 pages in length
  - What tests did you do on your compound? (instrumentation)
    - IR (record major peaks)
      - Compare experimental IR to starting material, as well as literature product IR
      - List any peaks that help in identification - 3-4 major peaks is a good rule,
      - Please attach relevant (labeled) IRs to your lab report.
    - TLC
      - Include developing solvents (i.e. 50:50 ethyl acetate: hexane), and visualization method
    - Melting point (how many trials?)
    - NMR
  - How do you know (using the tests) what your compound is?
    - Critically discuss your data
    - How does each piece of data narrow the possibilities of what it could be? Is the data contradicting at all?
  - Remember in your results and discussion you are trying to prove some basic points. Did your reaction work or not? Your results and discussion should be geared to answering this question. Some data collected may support or refute a particular finding, but it is important to discuss what each piece of data tells you about your experiment and why you came to that conclusion. Simply giving a bunch of results without explanation does not make for a very good results and discussion. Theoretically your results and discussion should account for half or more of your report.
  - Your experiment will not always work/ you will not always finish
    - See if you can work with a friend if you don't get any product. Discuss the analysis of the product together and use the shared information in each individual report.
    - Why do you think your experiment didn't work? Think chemically speaking, not just human errors.
- Conclusions (4 points)
  - One paragraph in length
  - Usually written in present tense
  - Make a stand – were you successful or not? Why or why not? Support it with your data (brief summarize)
  - Give some ways to improve the experiment next time.
- Grammar and spelling are just as important as the data itself. Great data is of no use to anyone if it is poorly presented.
- I expect you to use ChemDraw for structures

- Use school computers or download for your computer
  - ChemBioOffice is available at the SLC on both Windows and Mac computers. U of M also has a site license for ChemOffice Ultra which allows anyone with a U of M email address to download and install the software. To obtain the software, go to this URL: http://sitelicense.cambridgesoft.com/sitelicense.cfm?sid=157. There is also a link at the CTools site under "Resources".
- You must cite your procedure and where you get your constants/ chemical data from! See citation guide (end of this document) for desired citation format
- These sites might be useful for reference purposes (**Absolutely NO Wikipedia sources**):
  - NIST Chemistry Webbook - http://webbook.nist.gov/chemistry/
  - SDBS - http://riodb01.ibase.aist.go.jp/sdbs/cgi-bin/cre_index.cgi?lang=eng
  - Sigma-Aldrich - http://www.sigma-aldrich.com

GSI Points (***110 points, 11 labs at 10 points each)***

- GSI points will be assessed for all lab sessions starting from the second half of Expt. 1
- Points will be based on lab performance, safety infractions, lateness, failure to clean-up after oneself, etc. GSI points will not be based on yield or how quickly into the lab period you finish experiments.
- For Expts 5, 9, and 10, the quality of the lab notebook recording will also be taken into consideration.

Total points: 435 points

SOME TIPS ON LAB REPORT WRITING

- Please, please, please check your reports for spelling and grammar! Silly grammar and especially spelling mistakes really detract from your overall report. It's hard to take your results seriously if there are a lot of spelling or grammar errors. Use the spell check feature and always proofread your work!!
- Make sure to double-space your reports. I would rather have things be double spaced and go a bit over 3 pages, than have things single spaced and be under 3 pages.
- Do not use the first person tense in your lab reports, use third person past tense. "I" is unacceptable in formal scientific writing. You shouldn't even be using it in your lab notebook. Generally speaking, your introduction should be in the present tense, your experimental and results & discussion in the past tense, and your conclusion perhaps either one depending on how you do it. (A quick way to lose lots of points is to use "I"s everywhere)
- Write objectively, without adding personal qualifications to your analysis. Statements like "the experiment went pretty well" aren't appropriate for scientific writing. Don't write in a particularly negative fashion (i.e., whining), complaining about this or that in your conclusions or elsewhere in your lab report. Constructive means to improve the experiment are welcomed, but lack of resources or materials should not be used as reasons for why your particular experiment might have failed.
- Learn the proper abbreviations for amounts like grams (g), milligrams (mg), millimoles (mmol), milliliters (mL). Note that there is a space between the number and the unit…like 6 g.
- Find the actual degree sign (Insert, Symbol) and use it when giving a temperature, a space is used between the number and the degrees sign but not between the degrees sign and C…so 70 °C.

- All decimal numbers should have the leading zero before the decimal place…its 0.5 not .5
- Make sure you making the things that are supposed to be superscripted are superscripted (like the -1 in cm^{-1}, the units for wavenumbers) and the things that are supposed to be subscripted are subscripted (like the 4 in $MgSO_4$)
- Citations for your resources are very important. If in your introduction you use a great bit of material from the Ege text or elsewhere, you need to cite this information. This is especially important for IR spectrum taken from journal articles. I need the citation so I can look up the spectrum if I wish.
- The Reaxys database itself is not a resource. The data was collected by an individual and probably published in a journal somewhere. This article should be cited in addition to the database information.

CITATION STYLE

The ACS style is a standard method of citation in academic publications that originated with the American Chemical Society (ACS). The printed versions of the ACS style manual are entitled *ACS Style Guide: Effective Communication of Scientific Information*, 3rd ed. (2006), edited by Anne M. Coghill and Lorrin R. Garson, and *ACS Style Guide: A Manual for Authors and Editors* (1997).

Titles of journals are abbreviated; e.g.:

J. Am. Chem. Soc. – Journal of the American Chemical Society
J. Org. Chem. – Journal of Organic Chemistry
Org. Lett. – Organic Letters
Tetrahedron – Tetrahedron
Tetrahedron Lett. – Tetrahedron Letters
Acc. Chem. Res. – Accounts of Chemical Research

Article published in a journal:

Last Name, First Initial; Last Name, First Initial. *Journal*. **Year**, *Volume*, Pages.

Example of a journal citation:

Deno, N. C.; Richey, H. G.; Liu, J. S.; Lincoln, D. N.; Turner, J. O. *J. Am. Chem. Soc.* **1965**, *87*, 4533-4538.

URL (Web Page)

Author, if available. Title of page as listed on the site. Address of page (date accessed).

Example of Web Page:

SDBS: IR (Liquid Film), benzene. http://riodb01.ibase.aist.go.jp/sdbs/cgi-bin/direct_frame_top.cgi (accessed Apr 2011).

SOURCES: http://pubs.acs.org/books/references.shtml and http://www.cofc.edu/~chem/seminar/492/citing.html