424 Chemistry Lecture & Lab Information Sheet **Fall, 2016**

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Office Hours: M,W, 11-12 am (may still be in lab) & Tu,Th 4-4:50 pm

Lecture (24D-1224): Tu,Th 3:00-3:50pm (**CRN 72510**) Lab: #02 M,W 8:00-10:50pm (**CRN 73858**)
#01 M,W 3:00-5:50pm (**CRN 72511**)

Course Prerequisites Chem 316 (lecture, C-) and Chem 319 (lab, C)

Texts:

a) **The Organic Chem Lab Survival Manual, recent edition, James Zubrick**, This is a techniques book that you may have used in first year organic lab (required for Chem 317). If you did not sell your lab book back, you may still have it. Any recent edition is ok (7th – 10th).

b) **Miscellaneous Notes at Bronco Copy** – I will use my own Spectroscopy Book. It is available at Bronco Copy. It is about 370+ pages long and about $35. I used to use Introduction to Spectroscopy, (currently 5th edition) by Pavia, Lampman, Kriz and Vyvyan (new cost ~$305, used editions are less at Amazon). It is very well written and has plenty of problems (some answers are provided). If you need backup for my lectures, this is a student friendly book. It will cover advanced NMR topics using "real" examples, where my book uses "simulated" examples, though we will cover some of these methods in lecture. Pavia also covers UV spectroscopy It just seems too expensive (the 4th edition is pretty similar, . An even more advanced NMR text is NMR Spectrocopy Explained, by Neil Jacobsen (cost $138). If you are serious about learning most of the modern NMR experiments and instrumentation beyond our course, this is an excellent textbook.

c) We will use iclickers in lecture. You very likely already have one, but if not they can be purchased at the bookstore (new and used, either is ok). I think you can sell them back for half price when you are done at Cal Poly.

Tentative Schedule -

Lecture: Spectroscopic topics will be covered from my book and lecture notes. In lecture we will discuss MS and NMR methods and combination problems. IR will be introduced in lab, but problems will be included in lecture. A total of 75% of the grade will come from problem sets, quizzes, in class questions (clickers), and any other assigned work. A final exam will count for the other 25% of the lecture grade. It will cover all of the spectroscopy topics discussed in the course (lecture and lab).

Lab: There is a list of activities that are proposed, some as an entire lab and some at your own pace, in pairs. These activities will require strong self-motivation on your part to prepare, plan and work on. It is easy in the beginning to think that time is unlimited, since there is a semi-flexible schedule. However, if you work diligently from the start of the course, you may find that you are able to attempt most of the projects and that it is easier to relax at the end of the course while others are stressing out over unfinished work. You need to be an efficient and consistent worker to get as much done in each period as possible. A lab final will count 15% of the lab grade.

Method of Evaluation (the same grade will be assigned for lecture and lab, even though separate courses)

1. Lab Material (Notebook, Lab Projects, Unknowns, Quizzes, Problem Sets, Final Exam) 50%
2. Lecture Material (Problem Sets, Quizzes, Literature Assignments, iClicker Questions, Final Exam) 50%
3. An overall course grade will be based on the following percentages:

A 87-100 B 70-86 C 55-70 C- 50-54 D 40-49 F below 40

Final Exam Schedule (The lab final may be given on the last regular day of the quarter, after checkout)
Tu,Th, 3 pm, Lecture Final Exam Date.....Tuesday, Dec 6, 1:40-3:40 pm (bldg 24D – room 1224)
MW, 8 am Lab Final Exam Date.....Monday, 7:00-9:00 am (bldg 8 – room 312)
M,W, 3 pm Lab Final Exam Date.....Wednesday, 1:40-3:40 pm (bldg 8 – room 312)

Lectures: Tu, Th, 3:00-3:50, 24D-1224

Sep 20	No School
Sep 22	Elemental analysis, MW, Molec Formulas, types of spectroscopy and energy, (IR will be covered in the lab)
Sep 27	Lecture: Mass spec
Sep 29	Lecture: Mass spec
Oct 4	Lecture: NMR, what is it? Appearance of spectra (H+C), Instrumentation
Oct 6	Lecture: Chemical shifts (substituents and π bonds), different types of hydrogen and carbon atoms
Oct 11	Lecture: Predicting proton chemical shifts (sp3, alkene, and aromatic protons)
Oct 13	Lecture: Splitting patterns, J values, N+1 rule, when it works and when it doesn't
Oct 18	Lecture: predicting/interpreting ^{13}C chemical shifts
Oct 20	Lecture: Special types of NMR, (DEPT, COSY, HETCOR, NOE, HMBC)
Oct 25	Lecture: predicting/interpreting NMRs
Oct 27	Lecture: predicting/interpreting NMRs
Nov 1	Lecture: predicting/interpreting NMRs
Nov 3	Lecture: predicting/interpreting NMRs
Nov 8	Lecture: predicting/interpreting NMRs
Nov 10	Lecture: predicting/interpreting NMRs
Nov 15	Lecture: predicting/interpreting NMRs
Nov 17	Lecture: predicting/interpreting NMRs
Nov 22	Lecture: predicting/interpreting NMRs
Nov 24	Thanksgiving
Nov 29	Lecture: predicting/interpreting NMRs
Dec 1	Lecture: predicting/interpreting NMRs
Dec 6	Final Exam, 1:40-3:40

Lab: M, W, section 02, 8-11 am, section 01, 3-6 pm, 8-312

Sep 26	Check In, Safety Information, IR Lecture
Sep 28	Demonstrations, TLC/Flash Chromatography, Rotovap, TLC capillaries, NMR pipets, NMR demo, IR demo, KBr, Neat, Mull, Instrument, ATR platform, work on IR unknowns
Oct 3	various lab experiments and projects
Oct 5	various lab experiments and projects
Oct 10	various lab experiments and projects
Oct 12	various lab experiments and projects
Oct 17	various lab experiments and projects
Oct 19	various lab experiments and projects
Oct 24	various lab experiments and projects
Oct 26	various lab experiments and projects
Oct 31	various lab experiments and projects
Nov 2	various lab experiments and projects
Nov 7	various lab experiments and projects
Nov 9	various lab experiments and projects
Nov 14	various lab experiments and projects
Nov 16	various lab experiments and projects
Nov 21	various lab experiments and projects
Nov 23	various lab experiments and projects
Nov 28	various lab experiments and projects
Nov 30	Check out, Lab Final

Chem 424 Lab Projects (not all of the details have been worked out for these yet)

1. Safety questions and Lab Equipment questions must be answered before you can begin lab work. (20 pts total).

2. Identify possible IR unknowns from IR spectra and elemental analysis and molecular weight. 1. neat sample of a liquid and 2. KBr sample of a solid. Point out important frequencies in the spectrum which help you in the solution of your unknown and show your work to calculate a molecular formula from the give data. You will also need to turn in your lab notebook pages for the work done on this project. Someone else should be able to do the same work by just using your lab notebook pages. (10 points for each unknown, 10 points for your lab notebook pages = 30 total points). Turn in IR Tutor handout. (10 points)

3. Run a proton and carbon-13 NMR on your own, using instructions provided in handout (me watching). (20 points)

4. You will be given a two component mixture to be separated by simple and fractional distillation. You will need to run an IR, H-NMR, C-NMR on each component. Only run a GC-MS on the higher boiling component. Ask me to generate a MS for the lower boiling component. You will also need to turn in your lab notebook pages for the work done on this project. Someone else should be able to do the same work by just using your lab notebook pages. (20 points for each unknown and 20 points for your lab notebook pages = 60 total points)

5. You will be given a two component mixture to be separated by flash chromatography (determine solvent system using TLC, make spotting capillaries, collect fractions, determine which tubes have samples, strip off solvent using rotovap (or let evaporate at back of hood). You will need to run an IR, H-NMR, C-NMR and GC-MS (only if pure) on each component. You will also need to turn in your lab notebook pages for the work done on this project. Someone else should be able to do the same work by just using your lab notebook pages. (20 points for each unknown and 20 points for your lab notebook pages = 60 total points)

6. You will be given a two component mixture to be separated by extraction strategy designed by yourself using a strategy suggested in the text and/or handout to separate strong acid / weak acid / base / neutral components. You will have two of those possibilities. You will need to run an IR, H-NMR, C-NMR and GC-MS on each component. You will also need to turn in your lab notebook pages for the work done on this project. Someone else should be able to do the same work by just using your lab notebook pages. (20 points for each unknown and 20 points for your lab notebook pages = 60 total points)

7. You will have to isolate the natural product, eugenol, from cloves (follow the procedure given provided in the handout to isolate eugenol, (using steam distillation). You will need to run an IR, H-NMR, C-NMR and GC-MS on eugenol and provide a complete explanation to show how these confirm the structure. You will also need to turn in your lab notebook pages for the work done on this project. Someone else should be able to do the same work by just using your lab notebook pages. (20 points for your spectra and explanation and 20 points for your lab notebook pages = 40 total points)

8. You will use a Soxhlet extractor to isolate the natural product material (eugenol?) from cloves. You will need to run an IR, H-NMR, C-NMR and GC-MS on eugenol and provide a complete explanation to show how these confirm the structure. You will also need to turn in your lab notebook pages for the work done on this project. Someone else should be able to do the same work by just using your lab notebook pages. (20 points for your spectra and explanation and 20 points for your lab notebook pages = 40 total points)

9. You will have to follow the procedure from the damaged lab book of a previous research student (handout) and identify the product (relative stereochemistry too). You will need to run an IR, H-NMR, C-NMR and GC-MS on the product and provide a complete explanation to show how these determine the structure. You will also need to turn in your lab notebook pages for the work done on this project. Someone else should be able to do the same work by just using your lab notebook pages. (20 points for your spectra and explanation and 20 points for your lab notebook pages = 40 total points)

10. Use sublimation to get pure solid material from an unknown solid mixed with an inert solid material. Microscale kit.

11. Diol oxidation to an unknown product. Handout.

12. Naproxene isolation. Handout

13. If you finish all of the above work, then you are ready for graduate school or work in industry. Congratulations!

Handouts for Chem 424 course

1. Course Information Syllabus, calendars, lab projects
2. Lab policy, notebook example and grading scheme, list of lab activities
3. NMR instructions for running proton and carbon spectra
4. IR instructions
5. GC-MS instructions
6. Rotovap instructions
7. MSDS Information
8. Safety worksheet based on lab text
9. Miscellaneous other questions from lab text
10. Glove safety handout
11. Eugenol extraction handout
12. Unfinished experiment - glucose
13. IR unknown list (solid and liquid) – elemental analysis (or MS?)
14. Extraction Handout (acid/base)
15. IR Problem Sets and Examples
16. M-nova procedure (NMR files?)