CHEMISTRY 4346 – ADVANCED SYNTHESIS
Fall 2015

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Office Hours: after the class or by appointment

Objective: This course is designed to teach you various methods of synthesis, isolation, purification, and characterization of inorganic, organic and organometallic compounds.

Briefing: SH205 (MW 1:00 – 1:50 pm) **Lab:** CRB 205 (MW 2:00 – 4:50 pm)

Lab manual: **A copy will be provided**
Notebook: **Student Lab Notebook (Chemistry Spiral Bound 50)**
ISBN: 9781930882232 By: Hayden-McNeil

EXPERIMENTS and SCHEDULE

Note that the instructor reserves the right to adjust this schedule in any way that serves the educational needs of the students enrolled in this course

First half of the semester: **Organic**

1. **Synthesis of a Flavone** – Multistep synthesis of a classical heterocyclic system
2. **Sonogashira Reaction** – A palladium and copper co-catalyzed cross-coupling reaction using microwave reactor
3. **CSI@UTA** – Structural assignment of a reaction product using advanced analytical techniques
4. **Organocatalytic Aldol** – Using proline as a catalyst
5. **Synthesis of a Quinolone** – A thermal rearrangement of a vinylogous amide

Second half of the semester: **Inorganic**

1. Reactions of a metal-metal bond
   Cyclopentadienyliron dicarbonyl iodide
2. Coordination compounds of a chelating ligand
   Preparation of tris(2,4-pentanedionato)cobalt(III): $Co(acac)_3$
3. Reactions on a coordinated ligand
   Nitration of tris(2,4-pentanedionato)cobalt(III)

4 Synthesis of a fluorinated pyrazole
Synthesis of 3,5-bis(trifluoromethyl)pyrazole

5 Synthesis of a metal containing ring
Synthesis of silver(I) pyrazolete complex using 3,5-$(CF_3)_2$PzH

Sometimes, we may have to divide the students in the class to two or more groups in order to complete all the assignments in a timely manner and as noted below. We will let you know if this is necessary, which depends on the number of students in the class.

Group 1 Experiments	**Group 2 Experiments**	**Group 3 Experiments**
1	1	1
2	4	3
3	5	4
4	2	5
5	3	2

Must submit the laboratory report for the first experiment before starting the third one on your list, and follow the same routine for the other experiments (e.g., report for experiment two on your list is due before starting the fourth one, etc.).

Find a partner within your group for Inorganic - Experiments 4 (this experiment is done in pairs)

Procedures and Techniques:
General reference: There are a variety of laboratory manuals available that describe general techniques in experimental synthetic chemistry. Much of this is covered in the Organic Chemistry Laboratories that you have taken already and as such the manuals you used contain this information –**you are responsible for ensuring that you are familiar with experimental techniques**.

Further information on these techniques can be found in:

Introduction to Organic Laboratory Techniques, Pavia, Lampman and Kriz.

You are responsible for knowing the basics of all techniques used in this course.

1. Keeping a Notebook, p. 14-21.
2. Solvents, Heating Reaction Mixtures, Technique 1, p. 465
3. Filtration, Technique 2, p. 474.
4. Crystallization, Technique 3, p. 481.
5. Melting Point Determination, Technique 4, p. 492
6. Extraction, Technique 5, p. 500.
7. Distillation, Technique 7, p. 534-539.
8. Azeotropes, Technique 7, p. 534-539.
9. Measuring Pressure, Technique 9, p. 547.
10. Gas Chromatography, Technique 12, p. 585.
11. Preparation of Samples of Spectroscopy, Technique 17, p. 615.

SAFETY:

Mandatory Online Safety Training: Students registered for this course must complete the University's required "Lab Safety Training" prior to entering the lab and undertaking any activities. Students will be notified via MavMail when their online training is available. Once notified, students should complete the required module as soon as possible, but no later than their first lab meeting. Until all required Lab Safety Training is completed, a student will not be given access to lab facilities, will not be able to participate in any lab activities, and will earn a grade of zero for any uncompleted work.

1. You should have received an email from the UTA Compliance Department. Click on the link in the email (or navigate to https://training.uta.edu for the login page)
2. Log on using your network log-on ID and password (what you use to access email). If you do not know your NetID or need to reset your password, visit http://oit.uta.edu/cs/accounts/student/netid/netid.html.
3. The available courses for completion will be listed. For Chemistry 1441, complete the course entitled 'Student Lab Safety Training'
4. If you did not receive the training email and you have not already completed the training you will need to contact the training helpline (817-272-2080) or email compliance@uta.edu.
5. Students who have not completed the training by census date may be dropped from the lab (and consequently the lecture).

Once completed, Lab Safety Training is valid for the remainder of the same academic year (i.e. through next August) for all courses that include a lab. If a student enrolls in a lab course in a subsequent academic year, he/she must complete the required training again. **All questions/problems with online training should be directed to the University Compliance Services Training Helpline at 817-272-2080 or by emailing compliance@uta.edu.**

You will be exposed to hazardous chemicals in this class. Personal protective equipment (PPE) is necessary to protect your body. **Safety goggles for eye protection and a lab apron are required at all times in the laboratory.** Violation of this rule or other safety rules will lead to the loss of safety points and possibly dismissal from the laboratory period with a resulting grade of zero on the lab report. Goggles and aprons are available at the UTA Bookstore. Horseplay or other dangerous behavior will result in the immediate expulsion of the student for the remainder of the period with a resultant grade of zero on any related work.

Since the chemicals and equipment used in this course can be extremely dangerous if handled improperly, it is very important that you be completely familiar with the experiment (health, reactivity and fire hazards, etc.) before beginning it. Always use proper safety equipment and follow safe procedures.

In general, use your common sense, read the labels, be aware of your surroundings, keep your work area clean, never use open flames for heating (only oil baths and heat-guns are allowed) and properly dispose the waste. If you have any questions, ask the teaching assistant or instructor.

COURSE GRADE

1. Syntheses: 80%

a) purity *40% (assessed by mp, NMR spectrum, GC-MS)*
b) yield *40%*

Submit the properly labeled sample (include your name (and partner's name), experiment # and the compound name, % yield, m.p./b.p., etc)

You must first get the permission of the instructor, if you need to repeat an experiment. You will lose 15 points from the grade of the experiment for repeats.

2. Lab Reports: 20%.

A major component of a practicing scientist's time is taken up with writing – reports, grant proposals, etc., and so it is important that you learn how to write with accuracy and brevity. We will use templates for ACS publications so that you get some experience writing scientific articles – more details will be provided in initial briefing.

Layout of the lab report:

Title – self explanatory
Introduction – a brief description (one or two paragraphs) about why you are doing a particular experiment.
Results and Discussion – state the results, comments on whether the results are good, bad and what might be done to improve them, answers to questions.
Experimental – description of chemistry performed (use passive voice) include results (show yield, % yield calculation, m.p./b.p., interpret the spectra and assign key peaks, etc.),
References
Notebook – attach a copy from your lab notebook to the back of the report.

Letter grades:

Based on the average of the combined numerical values from organic and inorganic laboratory sections. Following numerical values approximate the letter grades which will be assigned

≥85	**A**
71-84	**B**
61-70	**C**
51-60	**D**
Below 50	**F**

NOTES

1. Each student is responsible for reading discussions of the basic techniques (distillation, crystallization, extraction, etc.). See Procedures and Techniques pages.
2. All syntheses are to be performed in the synthesis laboratory unless I give special permission to perform it elsewhere. Come to class on time.
3. Normally, syntheses are to be performed only in the scheduled laboratory periods. If additional time is needed on a scheduled day, you should notify the TA and arrange to come in early if possible.
4. Each product should be checked for purity using one or more techniques such as IR and/or NMR spectroscopy, GC-MS, melting point or boiling point. Melting points for solids and boiling points for liquids and % yields should be reported. Salt plates for IR and NMR tubes for NMR spectroscopy may be checked out from the stockroom. Attach all spectra to report sheets. An analysis (identification of major absorption peaks) of each IR and NMR spectrum should be made. Also, append the carbon copy from your notebook to the back of the report.
5. Keep your work area and equipment or other parts of the laboratory that you use (such as the balances, hoods, etc.) clean.

Attendance Policy: The following is from UT-Arlington Undergraduate Catalog's Academic Regulations section

Class Attendance
Class attendance and lateness regulations will be established by instructors and announced to their classes. At the discretion of the instructor, such regulations may or may not include provisions for making up work missed by the student as a consequence of an absence. Students who are late to class are responsible for reporting their presence to the instructor after the class is dismissed.

Information that stresses safety and technique is disseminated at the beginning of each lab period. Students are expected to be in the lab on time, and they will not be admitted to the lab more than 15 minutes after it begins. All missed work will receive zero credit. These 15 minutes are intended as a grace period for rare instances. It is not intended to become the norm. Abuse of this grace period will result in its cancellation.

Drop Policy: Students may drop or swap (adding and dropping a class concurrently) classes through self-service in MyMav from the beginning of the registration period through the late registration period. After the late registration period, students must see their academic advisor to drop a class or withdraw. Undeclared students must see an advisor in the University Advising Center. Drops can continue through a point two-thirds of the way through the term or session. It is the student's responsibility to officially withdraw if they do not plan to attend after registering. **Students will not be automatically dropped for non-attendance**. Repayment of certain types of financial aid administered through the University may be required as the result of dropping classes or withdrawing. For more information, contact the Office of Financial Aid and Scholarships (http://wweb.uta.edu/aao/fao/).

DROPPING:
• When dropping the course, YOU are responsible for seeing that all the proper paperwork is done by checking with the Chemistry Department office. If this paperwork is not completed - you will be given a normal letter grade corresponding to your earned grade - including Zeros for all missed work.
• YOU are also responsible for checking out of the lab. Failure to do so will result in your tuition bill being charged by UTA (charged as Chemical Breakage) plus amounts for anything that is broken, missing, or excessively dirty and a key replacement fee.

Academic Integrity: Students enrolled in this course are expected to adhere to the UT Arlington Honor Code:

> *I pledge, on my honor, to uphold UT Arlington's tradition of academic integrity, a tradition that values hard work and honest effort in the pursuit of academic excellence.*
>
> *I promise that I will submit only work that I personally create or contribute to group collaborations, and I will appropriately reference any work from other sources. I will follow the highest standards of integrity and uphold the spirit of the Honor Code.*

UT Arlington faculty members may employ the Honor Code as they see fit in their courses, including (but not limited to) having students acknowledge the honor code as part of an examination or requiring students to incorporate the honor code into any work submitted. Per UT System *Regents' Rule* 50101, §2.2, suspected violations of university's standards for academic integrity (including the Honor Code) will be referred to the Office of Student Conduct. Violators will be disciplined in accordance with University policy, which may result in the student's suspension or expulsion from the University.

Student Support Services: UT Arlington provides a variety of resources and programs designed to help students develop academic skills, deal with personal situations, and better understand concepts and information related to their courses. Resources include tutoring, major-based learning centers, developmental education, advising and mentoring, personal counseling, and federally funded

programs. For individualized referrals, students may visit the reception desk at University College (Ransom Hall), call the Maverick Resource Hotline at 817-272-6107, send a message to resources@uta.edu, or view the information at www.uta.edu/resources.

Disability Accommodations: UT Arlington is on record as being committed to both the spirit and letter of all federal equal opportunity legislation, including The Americans with Disabilities Act (ADA), The Americans with Disabilities Amendments Act (ADAAA), and Section 504 of the Rehabilitation Act. All instructors at UT Arlington are required by law to provide "reasonable accommodations" to students with disabilities, so as not to discriminate on the basis of disability. Students are responsible for providing the instructor with official notification in the form of a letter certified by the Office for Students with Disabilities (OSD). Students experiencing a range of conditions (Physical, Learning, Chronic Health, Mental Health, and Sensory) that may cause diminished academic performance or other barriers to learning may seek services and/or accommodations by contacting:
The Office for Students with Disabilities, (OSD) www.uta.edu/disability or calling 817-272-3364.
Counseling and Psychological Services, (CAPS) www.uta.edu/caps/ or calling 817-272-3671.

Only those students who have officially documented a need for an accommodation will have their request honored. Information regarding diagnostic criteria and policies for obtaining disability-based academic accommodations can be found at www.uta.edu/disability or by calling the Office for Students with Disabilities at (817) 272-3364.

Title IX: The University of Texas at Arlington does not discriminate on the basis of race, color, national origin, religion, age, gender, sexual orientation, disabilities, genetic information, and/or veteran status in its educational programs or activities it operates. For more information, visit www.uta.edu/eos. For information regarding Title IX, visit www.uta.edu/titleIX .

Students with Pregnancies: For students who are pregnant, it is recommended by the Chemistry and Biochemistry Dept. that you do not enroll into a chemistry lab at this time. If you become pregnant during the semester, we recommend dropping the course as soon as possible; and special provisions will be made to assist you in finishing the course at a later date. ***Please see your faculty instructor for assistance.***

P/F grade option: If P or F is a grade option in this class and you intend to take this class for a pass/fail grade instead of a letter grade, you MUST inform the instructor, through the necessary paperwork, of your intentions BEFORE the census date.

Electronic Communication: UT Arlington has adopted MavMail as its official means to communicate with students about important deadlines and events, as well as to transact university-related business regarding financial aid, tuition, grades, graduation, etc. All students are assigned a MavMail account and are responsible for checking the inbox regularly. There is no additional charge to students for using this account, which remains active even after graduation. Information about activating and using MavMail is available at http://www.uta.edu/oit/cs/email/mavmail.php.

Student Feedback Survey: At the end of each term, students enrolled in classes categorized as "lecture," "seminar," or "laboratory" shall be directed to complete an online Student Feedback Survey (SFS). Instructions on how to access the SFS for this course will be sent directly to each student through MavMail approximately 10 days before the end of the term. Each student's feedback enters the SFS database anonymously and is aggregated with that of other students enrolled in the course. UT Arlington's effort to solicit, gather, tabulate, and publish student feedback is required by state law; students are strongly urged to participate. For more information, visit http://www.uta.edu/sfs.

Final Review Week: A period of five class days prior to the first day of final examinations in the long sessions shall be designated as Final Review Week. The purpose of this week is to allow students sufficient time to prepare for final examinations. During this week, there shall be no scheduled activities such as required field trips or performances; and no instructor shall assign any themes,

research problems or exercises of similar scope that have a completion date during or following this week *unless specified in the class syllabus*. During Final Review Week, an instructor shall not give any examinations constituting 10% or more of the final grade, except makeup tests and laboratory examinations. In addition, no instructor shall give any portion of the final examination during Final Review Week. During this week, classes are held as scheduled. In addition, instructors are not required to limit content to topics that have been previously covered; they may introduce new concepts as appropriate.

Emergency Exit Procedures: Should we experience an emergency event that requires us to vacate the building, students should exit the room and move toward the nearest exit. When exiting the building during an emergency, one should never take an elevator but should use the stairwells. Faculty members and instructional staff will assist students in selecting the safest route for evacuation and will make arrangements to assist handicapped individuals.