CHEMISTRY 4346
Spring 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.

Lab manual: **A copy will be provided**

EXPERIMENTS

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 $3,5\text{-}(CF_3)_2PzH$

Organic

You have a copy already

Group 1	Group 2
1	1
2	4
3	5
4	2
5	3

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.

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%*
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.

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
Photo copies or originals of requested items

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**

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.

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, 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.
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.

Academic dishonesty: UTA considers academic dishonesty a completely unacceptable mode of conduct, and the University will not tolerate it in any form. Academic dishonesty includes (but is not limited to) cheating, falsification of data, plagiarism, and contracting/collusion with others to do your test or do your work. Cheating is the use or acquisition of information (data, constants, formulas,

textual material, etc.) from either unauthorized sources or in an unauthorized manner. If there is a suspicion of such activity your behavior will be reported to the Office of Student Conduct who will investigate and rule on the case.

Following is a statement from the University policy on cheating. *"Students who violate University rules on scholastic dishonesty are subject to disciplinary penalties, including the possibility of failure in the course and dismissal from the University."*

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.***

Americans with Disabilities Act (ADA): Students who qualify under the (ADA) should privately contact Prof. Dias or Dr. Lovely within the first week of classes to ensure that proper action will be taken to provide an acceptable classroom environment.

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.