CHEM 4461/CHEM 5461
INSTRUMENTAL ANALYSIS (Spring 2015)
LECTURE COURSE SYLLABUS

Instructor: Dr. Kevin A. Schug
CPB 358
817-272-3541
kschug@uta.edu (contact relating to miscellaneous items by email preferred)

Office hours: MW noon – 1 pm, stop-in, or by appointment

Text (required): Christian, Dasgupta & Schug *Analytical Chemistry*, 7th Ed.
Additional recommended texts for reference: Skoog, *Instrumental Analysis* and Harris, *Quantitative Analysis*
Additional resource: ChromACADEMY (www.chromacademy.com). A free registration is given for all University students for a period of five years.

Class Schedule:	Lecture:	SH 105	Section 001: M, W 11 – 11:50 am
	Lab:	CPB 215	Section 101: M, W 1 – 4:50 pm
		CPB 215	Section 102: T, Th 1 – 4:50 pm

Grading:	Exams	200 (2 x 100 pts.)
	Laboratory	800 (4 x 200 pts.)

Description and Goals of the Course: This course explores the fundamental basis of chemical analysis. It is designed to give the student a solid conceptual background and hands-on practice to understand how analytical techniques can be used to solve problems; including their limits and advantages. The emphasis is on solutions analysis and the course covers basic measurements and concepts, spectroscopy, chromatography, and mass spectrometry.

Student Learning Outcomes:

- Students can recall and apply knowledge gleaned from previous courses, especially Quantitative Analysis, and apply this to new analytical applications, especially the use of instrumentation.

- Students can explain general and specific concepts in liquid and gas chromatography. They can distinguish between applications where one or the other might be appropriate.
- Students can demonstrate the ability to choose appropriate instrumentation for solving various analytical problems and the ability to design sound experimental procedures.
- Students can diagram general instrumental components and hardware used in chromatography, spectroscopy, and mass spectrometry.
- Students can create common dissemination schemes for exhibiting the work they have performed in the instrumental laboratory.

Attendance: At The University of Texas at Arlington, taking attendance is not required. Rather, each faculty member is free to develop his or her own methods of evaluating students' academic performance, which includes establishing course-specific policies on attendance. As the instructor of this section, I allow students to attend class at their own discretion, but strongly encourage attendance.

Tests, Finals, and Grading: At least a 10-point grading scale will be assumed (e.g. 90-100 A; 80-89 B; etc.), however the instructor reserves the right to adjust this scale to accommodate the spread of grades in the course. All exams will be given on the date specified by the instructor (approximate dates are given at the end of the syllabus). All exams will be worth 100 points.

Tools for Success in This Course:

1. Attend class!! Complete assignments on time.
2. Keep up with material. Review expected learning outcomes and read relevant chapters before lecture. Formulate questions if a concept is unclear.
3. Consult multiple texts.
4. Dedicate appropriate study time. In Chemistry, you should consider spending three (3) hours studying outside of class for every one (1) hour of lecture.
5. Review your lecture notes after every class and seek to clarify any points which are unclear.
6. Don't procrastinate. These concepts take time and practice to sink in, so do not leave studying until the night before an exam.
7. Form a study group. Meet regularly to solve problems together and obtain help with difficult concepts. Collect contact info for each of your study group members.

Approximate Lecture Schedule:

Date (Day)	Material/Activity
1/21/15 (W)	No Class; Complete CHROMAcademy Modules: 1) The Theory of HPLC/Introduction 2) Theory and Instrumentation of GC/Introduction
1/26/15 (M)	Intro (syllabus); What remembered from Quant?
1/28/15 (W)	Introduction to chromatography
2/2/15 (M)	Basics of sample preparation
2/4/15 (W)	Basics of gas chromatography
2/9/15 (M)	Basics of liquid chromatography
2/11/15 (W)	Basics of mass spectrometry (flipped class)
2/16/15 (M)	Basics of molecular spectroscopy (flipped class)
2/18/15 (W)	Basics of atomic spectroscopy
2/23/15 (M)	Fluorescence and phosphorescence
2/25/15 (W)	Important parameters in gas chromatography
3/2/15 (M)	The van Deemter curve
3/4/15 (W)	**EXAM 1**
3/9/15 (M)	No Class (Spring Break)
3/11/15 (W)	No Class (Spring Break)
3/16/15 (M)	HPLC Separation Modes 1
3/18/15 (W)	HPLC Separation Modes 2
3/23/15 (M)	Mass Analyzers 1
3/25/15 (W)	Mass Analyzers 2
3/30/15 (M)	Electron ionization/chemical ionization
4/1/15 (W)	Atmospheric pressure ionization 1
4/6/15 (M)	Atmospheric pressure ionization 2
4/8/15 (W)	Advanced Topics 1
4/13/15 (M)	MALDI-MS (on-line)
4/15/15 (W)	Ion Mobility Spectrometry (on-line)
4/20/15 (M)	Protein Separations (on-line)
4/22/15 (W)	Protein Mass Spectrometry (on-line)
4/27/15 (M)	Review of On-Line Modules
4/29/15 (W)	**EXAM 2**
5/4/15 (M)	Advanced Topics 2
5/6/15 (W)	Advanced Topics 3

*I reserve the right to alter this schedule as necessary to ensure optimal instruction for the students in this course --KAS

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/).

Americans with Disabilities Act: The University of Texas at 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)*. 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 that disability. Any student requiring an accommodation for this course must provide the instructor with official documentation in the form of a letter certified by the staff in the Office for Students with Disabilities, University Hall 102. 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 is committed to upholding U.S. Federal Law "Title IX" such that no member of the UT Arlington community shall, on the basis of sex, be excluded from participation in, be denied the benefits of, or be subjected to discrimination under any education program or activity. For more information, visit www.uta.edu/titleIX.

Academic Integrity: Students enrolled all UT Arlington courses 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.

Lab Safety Training: Students registered for this course must complete all required lab safety training prior to entering the lab and undertaking any activities. Once completed, Lab Safety Training is valid for the remainder of the same academic year (i.e., through the following August) and must be completed anew in subsequent years. There are no exceptions to this University policy. Failure to complete the required training will preclude participation in any lab activities, including those for which a grade is assigned.

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, which is located at the end of the hall to the right, or out of the front of the building (to the left). 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 individuals with disabilities.

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.

Writing Center: The Writing Center, 411 Central Library, offers individual 40 minute sessions to review assignments, *Quick Hits* (5-10 minute quick answers to questions), and workshops on grammar and specific writing projects. Visit https://uta.mywconline.com/ to register and make appointments. For hours, information about the writing workshops we offer, scheduling a classroom visit, and descriptions of the services we offer undergraduates, graduate students, and faculty members, please visit our website at www.uta.edu/owl/.

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INSTRUMENTAL ANALYSIS (Spring 2015)
LABORATORY COURSE SYLLABUS

Laboratory Portion of the Course: There will be four problem-based laboratory experiments to complete. At minimum, three weeks (or six lab periods) are allotted to each experiment. Groups (2 – 3 persons per group) will rotate between experiments, so that each group will be working on a different lab on any given week. The schedule and laboratory experiments are at the end of this syllabus. Each lab experiment will be worth 200 pts and will have seven components, as below.

Pre-lab Quiz…	20 pts.
Requisition & Procedures form…	30 pts.
Pre-experiment Report…	30 pts.
Lab Report Project…	100 pts.
Good Laboratory Practice/Collegiality…	20 pts.

At the beginning of each lab experiment, a **Pre-Lab Quiz (20 pts)** will be administered to each individual. The quiz is meant to test your independent preparation for understanding fundamental concepts of the instrumental technique you will use. Pre-lab quiz will be given in the first 10 minutes of lab and each person must be present in order to take and receive credit for the quiz. No make-ups will be given without a documented excuse. A list of possible quiz questions is given at the end of the manual, to help you prepare for the quiz. Refer to the calendars below for quiz dates. Grades for Pre-Lab Quizzes are per individual.

Prior to beginning each experiment, you must fill out a **Requisition & Procedures Form (30 pts)** to request necessary chemicals and justify their need in the context of your designed experiment. A brief description of materials and methods planned should be given, along with cited references. A requisition form should be emailed by your group to your TA on the Friday (by 5 pm) indicated in the lab schedule for each lab experiment. A late penalty of 5 points per day will be assessed for requisition forms not received by the due date and time. Grades for Requisition & Procedure Forms are by group.

A short **Pre-Experiment (30 pts)** activity will be performed by each group on each instrument to familiarize you with the instrument. This will be done as part of the first lab period assigned for each experiment. The pre-experiment report will be submitted as a separate document (maximum 2 pages written, including exhibits and figures; single-spaced, 12 pt font, 1" margin) at the end of the second week of each experiment (Friday by 5 pm on the dates indicated on the schedule below). All submissions will be electronically to your TA in Word documents. The pre-lab should be given the file name: "4461preexpt_expt #_last name_first initial.doc". Points will be deducted by the TA for any deviation from this file name format. Grades for Pre-Experiments are given on an individual basis.

Each laboratory project is problem-based. While you will have defined instructions for the pre-experiment, no experimental design is given for the actual laboratory experiment. You will need to meet with your lab group (out of class/lab), and formulate an Experimental Design for each experiment. This may also include some considerable literature searching for appropriate methods. Overall, be sure to give proper consideration as to how you will address statistical aspects in your design, data collection, and presentation. Your experimental design must be accurately conveyed in your lab report projects. You are encouraged to consult with your TA and Dr. Schug as to the appropriateness of your experimental design. ***Begin the process of choosing and planning your laboratory experiment well before you attend lab. The time in lab should be spent doing your experiment. If you are not prepared, you will not have time to finish. Even the pre-experiments require a little background research (e.g., refer to old lab manual) in order to perform effectively. Once an experiment has been chosen and completed by one group in a section, it cannot be chosen by another group in any section.***

IMPORTANT: If you think you need to order any chemicals, propose your order to your TA, as soon as possible. Know a source (manufacturer), catalog number, price, and amount available/needed for each proposed order. Be able to justify your desire to purchase the chemical. In many cases, various chemicals can be obtained in-house; however, keep in mind that there are limits associated with how long it takes for ordered chemicals to arrive. USE REQUISITION FORM.

The outcomes of the experiment and how they are reported in your lab report project are important, but they do not dictate the majority of your grade. In other words, if things do not work out as hoped, then you can still get a reasonable grade if the experimental design, the presentation of the work, and other lab components are sound.

Presentation and style will be assessed for each lab report project. However, the formats for these **Lab Report Projects (100 pts.)** will vary, as described below.

1st Rotation Project Report: *JACS* Communication
2nd Rotation Project Report: Standard Operating Procedure (SOP) Document
3rd Rotation Project Report: Research Poster
4th Rotation Project Report: Vendor Application Note (publishable?)

The first rotation project report will be prepared in accordance with a submission to Journal of American Chemical Society Communications (http://pubs.acs.org/page/jacsat/submission/authors.html) . A template for the article submission can be found here: http://pubs.acs.org/page/jacsat/submission/jacsat_templates.html.
No table of contents graphics, keywords, author addresses, associated content (supporting information), or acknowledgements are needed. The submission is limited to three (3) template formatted pages total, including figures, tables, and references. You must include at least five (5) primary literature references to support your experiment and report. A maximum of four (4) tables and/or figures can be included in the manuscript. Significant care should be given to readability of text and exhibits, as significant penalties will be incurred if grammar, clarity, or format of the submission are compromised. Make sure that the tables and figures you choose are properly chosen representations of your experimental outcomes. ***Refer to published JACS Communication articles for inspiration and guidance on clarity and presentation.***

Grading rubric for JACS Communication:

- Title: 5 pts
- Abstract: 10 pts
- Introduction: 15 pts
- Method (written): 20 pts
- Experimental Design: 20 pts
- Results: 20 pts
- References: 10 pts

IMPORTANT: Individual reports from each group member should be submitted electronically in Word format to your TA, on the Friday following the week when the lab was completed. The dates when lab reports are due are listed in red on the laboratory schedule (below). The report should have the filename: "4461report_expt #_last name_first initial.doc". Points will be deducted by the

TA for any deviation from this file name format. ***Partners in groups can work together to design the experiments and collect the data, but each group member must prepare their own report, and consequently, figures and tables should not be identical. Deviations from this practice will be assessed for evidences of academic dishonesty.*** Late policy: For lab reports and pre-experiment reports, each will be assessed a 10% penalty (3 points for pre-experiment reports and 10 points for lab reports) per day that either is received after the due date.

The second rotation project report will be a Standard Operating Procedure (SOP). Pretend that you are working for a company that wishes to routinely carry out the laboratory experiment you devised. Prepare a detailed SOP that carefully guides the reader through the sample preparation steps (specific volumes, chemicals, concentrations, procedures, etc.). Use the data from your experiment as exemplary results. A comprehensive EPA guide to writing an SOP can be found here: http://www.epa.gov/quality/qs-docs/g6-final.pdf. Each individual of a group should write their own SOP.

Grading rubric for SOP:

- Title: 3 pts
- Application/ Scope: 8 pts
- Summary of Method: 8 pts
- Health and Safety Warnings/ Cautions: 8 pts
- Equipment and Supplies: 8 pts
- Sample Prep and Analysis Procedure: 25 pts
- Instrument Parameters: 10 pts
- Quality Control: 10 pts
- Results: 10 pts
- Overall Organization: 10 pts

The third project report will be a poster presentation. This will be done as a group. An electronic copy of the poster as a PDF will be submitted on the due date, and the poster will be presented during the designated class period. An example poster template will be provided.

Grading rubric for Poster Presentation:

- Organization: 20 pts
- Presentation/ Q&A: 30 pts
- Content: 40 pts
- Results: 10 pts

The fourth project report will be an Application Note. This should be similar to application notes created and distributed by instrument manufacturer vendors. In the case where the quality of the application note is high, it may be solicited to the instrument manufacturer (e.g., Shimadzu Scientific Instruments, Inc.) for publication. The application note will be created as a group; one App. Note will be submitted per group for grading.

High quality application notes will most likely be pitched to Shimadzu or BMG Labtech in the case of UV/vis plate reader. Visit http://www.ssi.shimadzu.com/literature/ for examples. You may need to sign up to view some of these. It's free.

Here are some examples, though not all contain all criteria listed below:
Thermal:
http://www2.shimadzu.com/applications/Thermal%20Analysis/Shimadzu_Thermal_T144.pdf

GC: http://www2.shimadzu.com/applications/gcms/C146-E260.pdf (brochure of many applications)

LC: http://www2.shimadzu.com/applications/LC/L442.pdf

UV/Vis: http://www.bmglabtech.com/en/applications/application-notes/

Grading rubric for Application Note:

Title: This should contain the application and the technique. (5 points)

Introduction: 3-5 Sentence paragraph on background of what is being analyzed and why the analysis is important. Have at least 4 citations here. Assume your readers are experienced operators of the instrument(s). (15 points)

Experimental: Describe sample preparation and instrumental parameters [have paramters in a table along with instrument make and model and column, if applicable] in sufficient detail that an experienced operator would be able to follow. Also describe quality control such as blanks and standards. (30 points)

Results: Your results should be displayed neatly.

Quality of results (10 points)

Neatly displayed results (20 points)

Conclusion/ summary: 3-5 sentence paragraph. (10 points)

Organization: The application note looks professional. Cite references in ACS format. App note should be 1-2 pages. (10 points)

DO NOT UNDERESTIMATE THE TIME IT TAKES TO PREPARE A QUALITY LAB REPORT PROJECT!

Finally, for each lab experiments, a score for **Good Laboratory Practice/Collegiality (20 pts)** will be levied by your TA. The awarding of these points is at the complete discretion of your TA. Points will be awarded based on whether you remained on task, were punctual, were collegial, etc.

Laboratory Schedule and TAs

Section	Day/Time	TA/email	Office/Ph	Office Hours
101	MW 1-4:50pm	Chandan Barhate (chandan.barhate@mavs.uta.edu)	CRB 315 2-1095	T 10 am – 12 pm; Th 2 – 4 pm
102	TTh 1-4:50pm	Ling Bai (ling.bai@mavs.uta.edu)	CPB 233 2-0618	W 1 -5 pm

Course Instructor: Dr. Kevin A. Schug (kschug@uta.edu), CPB 358

Week	Expt 1 (GC)	Expt 2 (Spec)	Expt 3 (HPLC)	Expt 4 (Materials)
1/21 W & 1/22 Th	**Check in for Section 101 (MW) & 102 (TTh)**			
1/26 – **1/30**	Group 1	Group 2	Group 3	Group 4
2/2 – 2/6				
2/9 – 2/13*				
2/16 – **2/20**				
2/23 – **2/27**	Group 4	Group 1	Group 2	Group 3
3/2– 3/6*				
3/16 – **3/20**				
3/23 – **3/27**	Group 3	Group 4	Group 1	Group 2
3/30 – 4/3*				
4/6 – **4/10**				
4/13 – **4/17**	Group 2	Group 3	Group 4	Group 1
4/20 – 4/24*				
4/27 – 5/1				
5/4 – **5/8****	**Check-out on 5/4 (Sec 101) and 5/5 (Sec 102)****			

- Specific dates highlighted in red indicate due dates for Lab Report Projects (2/27, 3/27, 4/17, and 5/8)
- Dates in green indicate dates for Pre-Lab Quizzes (2/2 or 2/3, 2/23 or 2/24, 3/23 or 3/24, 4/13 or 4/14, depending on lab section)
- An asterisk(*) indicates the dates that Pre-Experiment reports are due (2/13, 3/6, 4/3, 4/24)
- **Underlined dates** indicate due dates for Requisition and Procedure Forms (Template provided) to be submitted to your TA. The form must be approved

by your TA before you can begin the experiment. Requisition and Procedure Forms are due 1/30, 2/20, 3/20, 4/10.

- In the first week (1/21(W), 1/22(Th)), lab check-in, group assignment, and lab intro will be carried out. A comprehensive listing is below.
- ** - Lab Checkout/Cleanup; all must attend (5/4 (Sec 101) and 5/5 (Sec 102)).

Summary Listing of Important Dates and Lab Due Dates

Wednesday, 1/21/15: Section 101 (MW) check-in, group assignment, briefing
Thursday, 1/22/15: Section 102 (TTh) check-in, group assignment, briefing
Friday, 1/30/15: Requisition Form for 1st lab experiment due to TA
Monday, 2/2/15: Pre-Lab Quiz #1 for Section 101 (MW lab)
Tuesday, 2/3/15: Pre-Lab Quiz #1 for Section 102 (TTh lab)
Friday, 2/13/15: Pre-Experiment Report for 1st lab experiment due to TA
Friday, 2/20/15: Requisition Form for 2nd lab experiment due to TA
Monday, 2/23/15: Pre-Lab Quiz #2 for Section 101 (MW lab)
Tuesday, 2/24/15: Pre-Lab Quiz #2 for Section 102 (TTh lab)
Friday, 2/27/15: Lab Report Project (*JACS* Comm) for 1st Experiment due to TA
Friday, 3/6/15: Pre-Experiment Report for 2nd lab experiment due to TA
Friday, 3/20/15: Requisition Form for 3rd lab experiment due to TA
Monday, 3/23/15: Pre-Lab Quiz #3 for Section 101 (MW lab)
Tuesday, 3/24/15: Pre-Lab Quiz #3 for Section 102 (TTh lab)
Friday, 3/27/15: Lab Report Project (SOP) for 2nd Experiment due to TA
Friday, 4/3/15: Pre-Experiment Report for 3rd lab experiment due to TA
Friday, 4/10/15: Requisition Form for 4th lab experiment due to TA
Monday, 4/13/15: Pre-Lab Quiz #4 for Section 101 (MW lab)
Tuesday, 4/14/15: Pre-Lab Quiz #4 for Section 102 (TTh lab)
Friday, 4/17/15: Lab Report Project (Poster; electronic) for 3rd Expt. due to TA
Friday, 4/24/15: Pre-Experiment Report for 4th lab experiment due to TA
Friday, 5/8/15: Lab Report Project (App. Note) for 4th Experiment due to TA

Data storage will be either by CD±R (write once, read many), or by a dedicated new and EMPTY flash drive at the discretion of the TA. ***Any flash drive will have your initials on it and must be verified virus free by the TA before each lab it is used. Absolutely no exceptions allowed.*** The penalty will impact your grade for the experiment you're performing in the form of a 10% deduction after grading if you use a drive without TA verification. Current infection rates for student flash drives for viruses/Trojans/malware in general is running over 60%. Please download Microsoft Forefront Endpoint Protection/Antivirus from the UTA website and install it on your home computers if you do not have a current, active antivirus package. Use it to test your hard and flash drives weekly.

******IMPORTANT: Electronically record all of your method and data files each lab period on your own electronic media. Data and method files on instrument computers will not be retained.******

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 4461/5461, 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 (817-272-5100) or email compliance@uta.edu.
5. Students who have not completed the training by census date may be dropped from the lab (and consequently any linked 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 817-272-5100 or by emailing compliance@uta.edu.

Laboratory Experiments

Experiments: Each experiment lists a pre-experiment, which should be the first thing completed when you enter the laboratory to start a new experiment. Where multiple experiments are listed, your group may choose which laboratory experiment under each category that they wish to pursue, after the pre-experiment is completed. You should meet, discuss, and research your chosen experiment, well in advance of convening in the laboratory to perform it. Time is limited, and if you do not finish the experiment, no concessions can be made. *All of your group members must be present to perform a given experiment, unless prior approval is obtained from the TA or Dr. Schug.*

As a general reference, the old Chem 4461/5461 lab manual can be found here: https://mavspace.uta.edu:443/kschug/4461%205461%20lab%20manual%20F%202009/CHEM%204461%20Student%20Manual%20F2009_rev20090825.pdf

REFER TO THE OLD LAB MANUAL (LINK ABOVE) FOR HELP WITH PRE-EXPERIMENTS AND INSTRUMENT-SPECIFIC CONCEPTS

Experiment 1:
Gas Chromatography and Gas Chromatography – Mass Spectrometry

Pre-experiment

[Perform all pre-experiment analysis using manual injection]

Goal: Use butane to determine the dead time, in triplicate. Develop a temperature-programmed GC method to baseline separate a mixture of benzene, toluene, and xylene in less than 5 minutes and identify each component.

Procedure: Make sure the instrument is on, the carrier gas is flowing, and the computer is responding. Follow the procedure on the front of the GC to condition the instrument to be ready for use.

There is also a small manual in a folder with instructions for using the software. Each student will make three manual injections of butane to determine the dead time by using an isothermal separation method. Insert the syringe needle into the outlet of a butane lighter and press the gas release button on the lighter (**Do not strike the flint**). Withdraw the plunger on the syringe each time to the same amount, be it 1ul or 3 ul. Insert syringe, inject, withdraw syringe, and start run. Record the retention time of the peak for each repetition and calculate the average and standard deviation for each individual and for the group as a whole. This is the dead time for the column under this method.

Prepare a 1% v/v mixture of benzene, toluene, and xylene (BTX) with a solvent of your choice (dichloromethane and methanol work well). Reach the goal by using a temperature gradient separation program and changes in flow rate. [Consider: How does GC detect things? How do you decide the temperature program you want? How does flow rate affect the result?] Identify each component.

Important: Retain your BTX mixture to use as a quality control standard. Each time you return to the lab to use the instrument, run this sample to insure that you are obtaining consistent results.

Dilute a portion of your BTX sample to a 20 ppm v/v concentration in your chosen solvent. Using method settings consistent with what you developed on the GC, run your sample on the GC-MS. Set a reasonable scan range for detection of each analyte and set the solvent delay to avoid the high abundance solvent from reaching the detector. Ask your TA for details on these settings.

In your pre-experiment report, calculate the capacity factor for each compound, and the selectivity and resolution for each pair of compounds. Also include a copy of a representative chromatogram for GC and GC-MS, and details of your method. Be sure to report the results of your dead-time determination.

GC and GC-MS Experiments

[Once ANY GC or GC-MS experiment has been performed by ANY group in ANY class section, it may not be performed by any other group in any other section.]

GC.1) Characterize the retention of isopropanol, acetone, methyl-*tert*-butyl ether, benzene, and acetophenone using Kovats retention index.

GC.2) Compare the content of limonene in the peels of three different citrus fruits.

GC.3) Determine and compare the ethanol content of 3 different kinds of beer (i.e. lager, stout, IPA) by GC.

GC.4) Determine and compare the relative levels of ethyl alcohol in three different brands of mouth wash liquid by using gas chromatography.

GC.5) Design and carry out a selected ion monitoring experiment for the quantitative determination of methyl-*tert*-butyl ether and benzene in gasoline. Compare the content for two different gasoline samples.

GC.6) Develop a method to qualitatively assign and compare the presence of different terpenes and terpenoids in the peels of two different citrus fruits.

GC.7) Determine the identity of plasticizer components leached from a chloroform extract of three different plastic containers.

GC.8) Characterize and compare the hydrocarbon (linear, branched, aromatic, etc.) content of 3 liquid fuels (e.g. Coleman fuel, tiki torch fuel, kerosene, gasoline, and/or castor oil)

GC.9) Characterize and compare the polyaromatic hydrocarbon content of two samples of coal tar (e.g., from two different types of pavement).

GC.10) Qualitatively compare the aldehyde content of three different "hoppy" (e.g., India Pale Ale) beers by GC-MS.

GC.11) Devise an experiment to quantitatively evaluate the difference in sensitivity that can be attained by performing a splitless versus split (e.g., 200:1, 100:1, and 10:1) injection technique.

GC.12) Determine the relative levels of diacetyl (2,3-butanedione) in three different styles of lager beer using GC-MS.

GC.13) Construct a van Deemter plot with 10 different flow rates using toluene as a standard and nitrogen as a carrier gas.

GC.14) Characterize the chemical compounds that give rise to the scents of different flowers (at least 3 different flower varietals).

GC.15) Verify that a commercial pesticide spray (e.g. RAID wasp and hornet or other) contains the specified content of active ingredients.

Experiment 2:
Spectroscopy (UV-Vis, Fluorescence, Flame AA)

[You are expected to perform the pre-experiment for two out of the three spectroscopy instruments. Once ANY Spectroscopy experiment has been performed by ANY group in ANY class section, it may not be performed by any other group in any other section.]

UV-Vis spectroscopy; microarray plate reader

There is a very good manual with the instrument. Use it.

Pre-Experiment: Obtain one 96-well plate (you should use this through your pre-experiment and experiment until you run out of unused wells; each well should be used once; do not try to wash and re-use; keep careful track of the wells you used). Make a serial dilution (at least 9 points) of a 500 ppm solution of ethyl paraben in 50/50 water/methanol (v/v). Report a linear calibration curve based on the response of the compound at its maximum absorbance wavelength. Include a drawing (that you created) of a standard double-beam UV-vis spectrophotometer in your pre-experiment report. List three separate applications from the primary literature (provide references) for use of a UV-Vis microplate reader.

UV Experiments

UV.1) Develop and fully validate a method for the determination of acetophenone in contaminated well water. Full validation includes comprehensive determination of linearity, precision, accuracy, limit of detection, and limit of quantification. Be sure to research and include concessions for potential interferences.

UV.2) Determine and compare the levels of phosphate in three local bodies of water. In your report, speculate as to the origin of these compounds.

UV.3) Compare the relative levels of NADH and NAD+ in two different cell types or in two different species of bacteria.

UV.4) Develop a UV-spectrophotometric method for the simultaneous determination of aspirin and paracetamol in tablets.

UV.5) Using a UV-Vis microplate reader, determine the amount of food dye present in three types of hard candies (e.g. jolly ranchers). Also report the wavelength of the dyes present in the candies and make a qualitative comparison. Some hard candies may contain two or more food dyes.

UV.6) Compare the ability of three different sunscreen/sun block lotions to block UV light (i.e., compare magnitude and spectral range of their absorption properties). Determine the SPF value of the studied sunblocks and compare to the claimed value.

UV.7) Determine the phosphate content in three different types of soda.

UV.8) Measure and explain the temperature dependence of absorption spectral shifts for two different (vary the counter-cation) aqueous nitrate salt solutions. (An instrument for measuring T-dependent UV/Vis absorption is in the Shimadzu Teaching Instrument Lab).

UV.9) Measure the T-dependent auto-oxidation rates of quercetin. Determine rate constants at different temperatures and estimate the activation energy for the degradation reaction. (An instrument for measuring T-dependent UV/Vis absorption is in the Shimadzu Teaching Instrument Lab).

Fluorescence Spectroscopy

Pre-Experiment: There are 2 online manuals on the computer plus a helper manual at the instrument. Make a stock solution of 10 ppm quinine in 0.5% sulfuric acid. Develop a 7 point calibration curve from a series of dilutions of the stock solution. Ensure the calibration curve is in linear response range of the instrument. The maximum detector value is 1000 AU. Keep the response of the standards below 10% of this value.

System Configuration: Set slit bandpass to 2.5 nm, and the integration time to 0.5 sec. Repeat with slit bandpass of 10.0 nm. Report any differences, limitations of the two slit widths.

Prepare and report a calibration curve using the primary and secondary excitation and emission wavelengths (be sure to report the values used.) How do you find the excitation wavelengths to use? Include a drawing (that you created) of a standard fluorescence spectrophotometer in your pre-experiment report.

Fluorescence Experiments

FL.1) Determine the fluorescence properties (absorption/emission profiles, quantum yields, and limits of detection) for a series of biocompatible, photoluminescent polymers.

FL.2) Using fluorescence spectroscopy, determine and compare the levels of vitamin B2 in four varieties (varying manufacturers, milk-fat %, etc.) of commercial milk samples.

FL.3) Use fluorescence spectroscopy to determine the rate constant (kq) for oxidative quenching (electron transfer (eT) out to an electron acceptor), reductive quenching (eT in from an electron donor), and energy transfer (ET to a dye or other compound) for excited Ru(Phen)3 in solution. This analysis is best done using a Stern-Volmer plot. Potential quencher reagents to investigate are as follows:

Oxidative quenching: methyl viologen, Cu^{2+}, or Fe^{3+}
Reductive quenching: ascorbate ion (pH 5)
Energy Transfer: nickel (II) acetylacetonate (in MeOH)

FL.4) Compare absorption/emission profiles using UV-Vis and fluorescence spectroscopy for chlorophyll extracted from three different plants (Spinach, Tree, Grass, etc.).

FL.5) Determine the rate of lactate production from pyruvate and the enzyme lactate dehydrogenase by fluorometric spectroscopy.

FL.6) Use fluorescence spectroscopy to determine and compare concentration levels on riboflavin in a variety of vitamin pills

Flame Atomic Absorption Spectroscopy

Pre-Experiment: There is a folder containing startup information for the AA at the instrument. Prepare a set of calibration standards (minimum 7 different concentrations) ranging from 1.0 to 100 ppm (~15 mL each) Fe in 0.10 M HCl from a Fe compound in the lab. Generate a 1000ppm stock solution. Also prepare a blank of 0.10 M HCl (no Fe). Run and report a calibration curve for Fe in triplicate. Use the blank-corrected response on the x-axis. Include a diagram (that you created) for a flame AA instrument in your pre-lab report. Include a paragraph comparing and contrasting (advantages and disadvantages) flame AA, graphite furnace AA, and inductively-coupled plasma – atomic emission spectroscopy, as methods for determining metals in solution.

Flame AA Experiments

AA.1) Using flame atomic absorption spectroscopy, determine and compare the amount of copper in three varieties of mineral supplements.

AA.2) Determine and compare the levels of sodium in three different microwave popcorn varieties.

AA.3) Determine the copper content in different age U.S. coins of all common denominations by AA (e.g. compare dimes from pre- and post-1965; pennies from pre- and post-1982).

AA.4) Using flame atomic absorption spectroscopy, determine the amount of zinc present in a Cold-Eeze lozenge and compare with listed ingredients.

AA.5) Using flame atomic absorption spectroscopy, determine and compare the amount of copper, iron, magnesium, and zinc in each group member's hair.

AA.6) Using flame atomic adsorption spectroscopy, determine and compare the amounts of zinc and lead in three different soil samples.

AA.7) Using flame atomic adsorption spectroscopy, quantify the amount of calcium in 3 different fruit juices.

Experiment 3:
High Performance Liquid Chromatography, Liquid Chromatography – Mass Spectrometry, and Mass Spectrometry

[You should perform both pre-experiments listed]

Pre-Experiment 1 (HPLC-UV)

Goal: Develop a gradient reversed phase HPLC method for the separation of four parabens (methyl-, ethyl-, propyl- and buryl-paraben), using a C-18 column and a methanol/water mobile phase, in less than 7 minutes.

Procedure: Make sure the instrument is on, the two solvent reservoirs are filled and degassed, the computer is responding. Record the dimensions, the make and model, the type of the stationary phase, and the type of packing for the column installed in the chromatograph.
There is a small manual at the HPLC with software instructions.

Obtain a stock solution, which contains methyl-paraben, ethyl-paraben, propyl-paraben and butyl-paraben, each at a concentration of 50 ppm, in 50/50 methanol/water from your TA. Set the flow rate of the column to value suggested by TA (This is column dependent). Set the solvent ratio of the mobile phase to 99% methanol to 1% water, and turn the pump on. Set the analysis time to 8 minutes and the detector wavelength to 254 nm. Allow at least 5 column volumes of solvent to pass through the column before beginning the analysis. Note the pressure of the system and make sure it is stable prior to the run. Place sample in auto sampler. Note position in tray. Record the chromatogram and the peak analysis. Repeat this analysis sequentially for different mobile phase compositions (ex. 90/10, 70/30, 50/50, etc) until you have an idea how mobile phase affects the result, and you get baseline separation of the compounds. Remember to allow sufficient time for equilibration at each new mobile phase. These are isocratic separation methods.

Set up the instrument to perform a gradient solvent program separation method. Start with an initial mobile phase composition of 50/50 methanol/water, a hold at that composition for 1 min, an increase to 99/1 methanol/water in 6 minutes and a 1 minute hold at the final composition. Inject the 50 ppm mixture and record a chromatogram. Modify the solvent gradient to create a method for base line

separation (R>1.5) of all components in a time faster than your best isocratic separation.

Important: Retain your sample mixture and save your optimum gradient method to use for quality control each time you go back to the instrument. i.e., Run this sample under these conditions the first time you use the instrument in each lab to make sure it is working properly.

In your pre-experiment report, calculate the capacity factor for each compound and the selectivity and resolution for each pair of compounds. Also include a copy of your chromatogram and details of your method.

Pre-Experiment 2 (LC-UV-MS)

Obtain a mixture of caffeine and ibuprofen from your TA. Perform a separation (C18) of the mixture using three different mobile phase systems as specified by the TA: one with pure water and methanol, one containing an ammonium acetate modifier, and one an acetic acid modified. Compare UV and MS (positive and negative ionization mode) responses and peak shapes for each of the analytes under these conditions.

HPLC, LC-MS, and MS Experiments

[Once ANY LC or LC-MS experiment has been performed by ANY group in ANY class section, it may not be performed by any other group in any other section.]

LC.1) Develop a HPLC-UV separation method for a mixture of 4 hormone and endocrine disruptor compounds, and assign the identity of each peak in the chromatogram. Apply your method to determine the identity and concentration of a contaminated water sample.

LC.2) Develop an HPLC-UV method for separation and detection of anthocyanins from freeze-dried berries (at least three varieties). Compare and contrast the number and relative amount of anthocyanins in each of the berries tested. Using information from the literature on anthocyanins, attempt to assign the identity of each anthocyanin detected in each variety of berry you tested.

LC.3) Develop an analytical HPLC method for separating and quantifying caffeine and vitamin B6 in four different brands of energy drinks (Red Bull, Monster, Rock Star, etc.). Show a complete validation that includes linearity, precision, accuracy, limit of detection, and as well as limit of quantification

LC.4) Develop a method to determine pesticide content in three different varieties of tomatoes by HPLC. Target herbicides would be urea herbicides (tebuthiuron and diuron) and triazine herbicides (simazine, atrazine, and ametryn).

LC.5) Determine and compare the LOD and LOQ of caffeine and ibuprofen mixture using mass spectrometer vs. PDA.

LC.6) Develop and apply an HPLC-UV method for the detection of preservatives such as benzoic or sorbic acids in three different types of food items. (Possible food items include soft drinks, canned fruit/vegetables, sauces, jams/jellys, or dried fruit.)

LC.7) Systematically evaluate and report the effectiveness of selected ion monitoring vs. multiple different scan ranges (i.e., increasing magnitude of m/z scan ranges) on sensitivity and linear range for the LC-MS determination of quinine in tonic water.

LC.8) Design a flow injection analysis experiment to comprehensively evaluate the concentration dependent ionization suppression or enhancement effects of ammonium acetate, ammonium formate, acetic acid, and formic acid on the response of butyl amine in a 50/50 water/methanol solution.

LC. 9) Construct a Van Deemter plot with at least 10 different flow rates from isocratic separations of the caffeine and ibuprofen mixture.

LC. 10) Develop a HPLC-UV method for the extraction and separation of phenolic compounds, mainly flavanols and phenolic acids, from a variety of tea leaves (at least 3). Using a primary reference source, try to determine the identity of each phenolic detected and compare the relative levels of each phenolic in each tea.

LC. 11) Perform a qualitative and quantitative analysis of capsaicinoids in three different hot sauces.

LC.12) Develop a method to compare the relative content of photosynthetic pigments in three different plant varietals.

LC.13) Compare the anthocyanin identity and content of three different red flowers.

Experiment 4:
Materials and Thermal Analysis

[Once ANY Materials/Thermal Analysis experiment has been performed by ANY group in ANY class section, it may not be performed by any other group in any other section, until all of the experiments have been performed once.]

Differential Scanning Calorimetry (DSC)

Pre-Experiment

Goal: Develop a method of determining the melting point of a compound, and measure various values associated with the melting point using the analysis package.

Instrument Prep: There is a basic manual provided for this instrument. Please use it to properly set up this instrument. Be sure to turn on the Nitrogen gas prior to any use of the instrument. Not doing this can damage the instrument electronics. Before loading a sample, be sure that the internal temperature reading in software reads ≤ 40 °C.

Procedure: Determine the melting point of a small piece of Indium metal. Carefully cut a piece of Indium from the provided sample, trying to keep the weight between 3 and 7 mg using the analytical scale (analytical in this case means accurate to below 1mg).

Perform an experiment heating the metal from ambient to 200 °C at 10 degrees per minute. Using the analysis software described in the manual, measure the melting point and the amount of energy required per mg to melt the metal.

Now do the same for Zinc, going from ambient to 500 °C and report the melting point and amount of energy required per mg to melt the metal.

There are other transitions a DSC can identify. Please research these in your quest to find experiments for this instrument and mention them in the pre-lab, along with the presentation of results from the above experiments.

**A couple of important notes about our DSC:

- We have a "heat flux" DSC, which a bit different than the more common "heat flow" (aka power compensated) DSC. This website (http://www.colby.edu/chemistry/PChem/lab/DiffScanningCal.pdf) does a good job of describing DSC in general, but it refers to heat flow DSC. For heat flux DSC, all of the signals will be in the opposite direction. The difference between the two is given here: https://mavspace.uta.edu:443/kschug/2014%20spring%20Chem%204461/DSC_power%20Compensated%20vs%20heat%20flux.pdf
- Our DSC can be used to achieve temperatures as low as -140 ^{0}C. Ask your TA for details, but in order to operate it, you will need to run a Dry Gas (Ar or N_2) at a rate of 100-200 mL/min.

DSC Experiments

DSC.1) Determine and compare the heat capacities, glass transition temperatures, and melting temperatures for a series of polystyrene polymer standards, ranging in molecular weight from 2,500 to 50,000 Da.

DSC.2) Using DSC, evaluate the thermal events associated with the thermal degradation of aspirin. Particularly, investigate the effects of multiple heating and cooling cycles on the particular temperatures associated with these events.

DSC.3) Using TGA/DSC, study and compare thermal events associated with heating/cooling of three different varieties (e.g. densities) of commercial polyethylene polymers (e.g. milk jugs, juice bottles, etc.)

DSC.4) Compare and contrast the thermal events associated heating/cooling three different commercial plastic products produced from different polymers (e.g. PE, PP, PET, PC, etc.)

DSC.5) Compare and contrast polymorphic aspects of different samples of acetaminophen.

DSC.6) Investigate the retention of water by soil as a function of soil age. (e.g., vary the amount of time (up to days) that water is equilibrated with soil before it is analyzed).

Thermogravimetric Analysis

Pre-experiment

Turn on Nitrogen Gas and ensure gas is flowing before using the instrument! Not doing this can damage the instrument electronics

Goal: Obtain thermograms for the dehydration of copper sulfate pentahydrate and determine the weight percent of one molecule of H_2O by using formula mass.

Procedure:

Before loading a sample, be sure that the internal temperature reading in software reads ≤ 40 °C. Open the furnace; insert the protection plate so that you don't drop the pan into the furnace. **[Those pans are very expensive, if you drop them, you fail the experiment and you pay for the damage!]** Carefully put a secondary pan on the loading tray and close the furnace. Press Auto Zero button on the front of the TGA and wait for the instrument to complete the tare cycle. When it's balanced, open the furnace and take out the secondary pan to load your sample (enough to make a single uniform layer at the bottom of the pan) Carefully return the pan to the loading tray and make sure to align the pan in the middle of the tray. Close the furnace.

Perform an experiment of heating the sample from ambient to 300 °C at 5 degrees per minute and then another experiment with fresh sample going from ambient to 300 °C at 25 degrees per minute. **Do not begin another run until the furnace has cooled completely!** For each transition in the thermogram, determine and report the total and individual changes in the weight percent of your analyte. Determine and report the onset temperature for each transition. Compare the results of using different temperature ramps for the analysis. Include a copy of your thermogram with details of your method in your pre-experiment report.

TGA Experiments

TGA.1) Perform a "proximate analysis" for three different types of coal, and compare their properties.

TGA.2) Obtain four different samples of textiles and compare their thermal properties using TGA and DSC. Present the report for this analysis in the context of potential use of the method for forensics investigations.

TGA.3) Determine and compare the mass of hydrocarbon grafted onto silica phases used in HPLC column packings.

TGA.4) Obtain three different starch products and compare their thermal properties using TGA and DSC. Discuss how their properties relate to their use in food preparation.

TGA.5) Develop and validate a method to differentiate cobalt (II) carbonate hydrate from calcium sulfate dihydrate.

POSSIBLE PRE-LAB QUIZ QUESTIONS:

Spectroscopy

- Draw a schematic of a UV-Vis spectrophotometer
- Explain the difference between a single beam and double beam spectrometer.
- Draw a schematic of a fluorescence spectrophotometer
- Draw a schematic of a flame atomic absorption spectrophotometer.
- Why is hexane a good solvent for performing measurements by UV-Vis spectrophotometry?
- Give Beer's law and define each variable.
- What is the approximate wavelength range associated with ultraviolet light?
- Is visible light higher in energy or lower in energy than ultraviolet light? Explain.
- With respect to instrumental set-up why is fluorescence spectrophotometry more sensitive for the trace measurement of a fluorescent molecule compared to UV-Vis spectrophotometry?
- How does the energy of a fluorescence emission photon compare to the corresponding excitation event? Explain.
- What is the role of external conversion or quenching in the fluorescence process?
- What is a major limitation of flame AA in terms of monitoring the concentration of a wide range of metals in a sample?
- Why do atomic absorptions appear as sharp lines rather than the broad lines that are observed in molecular absorption spectroscopy?

Gas Chromatography and Gas Chromatography – Mass Spectrometry

- Draw a general diagram of a gas chromatograph instrument and label all parts.
- What are typical carrier gases for GC and what are their advantages and limitations?
- What type of detector is used for your GC experiments and what types of chemical compounds can it detect?
- What sample will you measure for determination of Kovat's Retention Indices?
- Define what is meant by the term "dead time" (t_0)?
- What parameter is calculated to determine whether or not you have achieved baseline separation of your analytes of interest?
- What is the role of polydimethylsiloxane in open tubular gas chromatography?
- What is a split ratio?
- How does electron ionization work?
- Why is electron ionization considered a hard ionization technique that creates odd-electron ions?
- How does a quadrupole mass analyzer work?

High Performance Liquid Chromatography and Liquid Chromatography – Mass Spectrometry

- Explain the mechanism of separating compounds by reversed phase liquid chromatography.
- What is meant by the term "isocratic separation"?
- How many analyte components will you have to separate in your HPLC pre-lab experiment? What is the order of expected elution for the compounds?
- Draw and label a general diagram of an HPLC instrument.
- Why is the UV cut-off of a solvent and important for mobile selection in HPLC-UV analysis? How is UV cut-off defined for a given solvent?
- Draw and label a general diagram of an electrospray ionization – mass spectrometer.
- What limitations are placed on additives to the mobile phase when you combine liquid chromatography with electrospray ionization – mass spectrometry?
- Describe the general mechanism of ion generation by electrospray ionization.
- Why is electrospray ionization considered a soft ionization source that produces even electron ions?

Differential Scanning Calorimetry and Thermogravimetric Analysis

- Explain how Differential Scanning Calorimetry differs from Isothermal Titration Calorimetry.
- Draw a general schematic of a DSC instrument.
- Explain how thermogravimetric analysis works.
- Draw a sample TGA thermogram and label pertinent features
- Draw an example DSC output plot and explain the meaning of positive and negative peaks observed.
- Explain why it is critical to avoid direct contact with any component of the DSC and TGA, including the sample pan.
- Explain how DSC might be used to characterize liquid crystal samples.
- Explain how TGA can be used to evaluate the thermal stability of a material.
- In terms of experimental technique, why is it possible to observe mass loss using TGA but not with DSC?
- Explain how a DSC sample should be prepared.
- What is added to the reference cell during a typical DSC experiment?
- Why is an inert gas such a N2 typically used during a TGA experiment?
- Describe how the loss of water would appear in a TGA thermogram.

<u>Want some extra credit?</u>

Students can suggest new lab experiment problems to be incorporated into future lab courses. Ask your instructor for details. Maximum two lab problem suggestions to be considered for each student for extra credit during the semester.

Other Important Items

Dropping: When dropping the course, YOU are responsible to see that the proper paperwork is filed with the Department. Failure to do so will result in a grade of "F".

Drop for non-payment of tuition: If you are dropped from this class for non-payment of tuition, you may secure an Enrollment Loan through the Bursar's Office. You may not continue to attend class until your enrollment loan is applied to outstanding tuition fees.

Grade Replacement: Students enrolling in this course with the intention to replacing a previous grade earned in the same course must declare their intention to do so at the registrar's office by Census Date of the same semester in which they are enrolled.

Pass/Fail: 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, before the Census Date.

Bomb Threat Policy: In the event of a bomb threat to a specific facility, University Police will evaluate the threat. If required, exams may be moved to an alternate location, but they will NOT be postponed. UT-Arlington will prosecute those phoning in bomb threats to the fullest extent of the law.

Americans with Disabilities Act: The University of Texas at Arlington is on record as being committed to both the spirit and letter of federal equal opportunity legislation; reference Public Law 93112-The Rehabilitation Act of 1973 as amended. With the passage of new federal legislation entitled American with Disabilities Act-(ADA), pursuant to section 504 of The Rehabilitation Act, there is renewed focus on providing this population with the same opportunities enjoyed by all citizens.

As faculty members, we are required by law to provide "reasonable accommodation" to students with disabilities, so as not to discriminate on the basis of that disability. Student responsibility primarily rests with informing faculty at the beginning of the semester and in providing *authorized* documentation through designated administrative channels.

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.
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 a 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.

Academic Integrity: All 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.

ALL PRIOR LAB REPORTS FROM PREVIOUS SEMESTERS ARE RETAINED FOR REFERENCE BY THE COURSE INSTRUCTORS. ELECTRONIC PLAGIARISM TOOLS ARE ALSO CONSULTED REGULARLY.

DO YOUR OWN WORK!