



CHEM 3472-002 (Fall 2016)

Instrumental Analysis

Term: Fall 2016

Lecture: TTh 12:00 – 1:15 pm; SLC 2.304

Laboratory: TTh 1:15 – 3:45 pm; BE 2.330

Course description:

The goal of this course is to provide you with practical experiences of using modern analytical instrumentation including ultraviolet, visible, fluorescence, atomic and mass spectroscopy, electrochemistry, surface and microanalysis, and chromatography. Emphasis will be placed upon fundamental principles, data acquisition and analysis, and report writing.

Learning Objectives:

Core Objective 1: This course is designed to prepare students for employment and/or further work in chemistry by providing instruction in methods of instrumental analysis, including chromatographic, spectroscopic, and statistical techniques.

Core Objective 2: This course is designed to help students gain a deep understanding of fundamental principles used in the design of a variety of analytic instrument through hand-on experiences.

Core Objective 3: Students will also learn how to keep a laboratory notebook with an emphasis on communicating procedures, observations and data in such a way that results could be duplicated. In addition, students will document data and observations in laboratory reports, develop skills for critical thinking, analyzing the data and drawing conclusions.

Core Objective 4: Students will also develop team-work attitudes including the ability to consider different points of view and to work effectively with others to support a shared purpose or goal.

Instructor:

Dr. Warren J. Goux

Office: BE3.510

Phone: 972-883-2660

Email: wgoux@utdallas.edu

Office hours: F 1-2 pm or by appointment

Other information: Use of standard computational programs, such as Excel, will be required.

Contact the instructor early in the course if assistance is needed.

Teaching Assistants:

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Prerequisite:

Prerequisite: CHEM 2401 Quantitative Analysis

Required Texts & Materials:

Principle of Instrument Analysis, 6th edition(2007) Skoog, Holler and Crouch: ISBN-13:978-0-495-01201-6

Recommended: Quantitative Chemical Analysis, 6th edition or later, Daniel C. Harris: Will place on reserve in the library

Lab Book: "Roaring Springs Composition Book", Quad. Rules 5 to 1", Bar Code 71072 77255, available in UTD Bookstore OR any other quad. Ruled notebook(required)

Homework:

Problems are designed to help you understand the fundamental principles of various analytic techniques. Similar problems might appear in the exams. You should turn in your homework one week after the work is assigned at or before the beginning of class time (<10 min after beginning of class). No late homework will be accepted. **All work must be turned in as a paper copy unless otherwise specified. No email submissions will be accepted.**

Quizzes:

From time to time we may have in class quizzes. These will count towards your grade the same as a homework assignment. Quizzes will be announced in class one week prior to the due date.

Exams:

Two 1-hour 15 min exams and one final exam will be given throughout the course. The dates of these exams will be confirmed in class and on the eLearning. All three exams must be taken. Your lowest hour 15 min exam score will be replaced by your score on the final exam if the final exam score is higher.

Labs:

TTh: 1:15 – 3:45 pm; Room: BE2.330

Before you begin an experiment you must have completed a prelab in your lab notebook. The TA will check your notebook to make sure your prelab is completed. All data must be entered into your notebook. Your notebooks must be signed at the end of the period by the TA or instructor. You must be present for the FULL lab period in order for the TA to sign your lab book and for you to be able to write up the experiment. There are no make-up labs.

Please visit <http://www.utdallas.edu/chemistry/resources/safety.html> for detailed safety rules

Lab Safety:

IMPORTANT: In accordance with University and Chemistry Department safety rules, any time anyone (student, TA, instructor, or visitor) is in a lab, Z87-rated safety eyewear must be worn. The first violation in the semester will result in a warning and removal from the lab until the safety eyewear is in-place. The second violation in the semester will result in dismissal from that lab period with no extra time being allowed for make-up of the work scheduled for that lab period. Similar penalties will apply if any other safety rules are violated. Please visit <http://www.utdallas.edu/chemistry/resources/safety.html> for detailed safety rules.

In addition, please refer to the supplemental handout concerning optical and electrical safety issues.

Notebooks:

You are expected to record all data in your notebooks. Each student must keep his or her own neat and orderly lab notebook using pen. You must bring your lab notebook during the lab periods. Please put your name and a date on every page you use. In addition, be sure to include data labels and units on all tables and graphs. Drawing chemical structures and balanced chemical reactions in your notebook is highly encouraged. **Your notebook must be signed and dated by your TA at the end of any day you are working in the lab. There are no make-up labs.**

We will evaluate each student during the lab period. Each student will be evaluated with respect to their lab performance: adherence to good safety practices, laboratory technical skills, and laboratory etiquette/professionalism.

Reports:

While you collect data with your lab partner, you must complete your own reports including graphs, calculations and answers. You should turn in your reports after the lab is conducted in a designated box on the second floor of Berkner (**outside BE 3.510**; see Appendix C for schedule). **An electronic copy should be turned in to the Turnitin box in eLearning.** The TAs will distribute detailed grading policies. **I will not accept email submission nor will I give you credit for turning it in on email. Only the paper copy is graded and this is picked up in the designated box in BE at 5:00 pm on Fridays. The electronic stamp given on eLearning will not count as the turn in time and the electronic copy is never graded. Grading errors should be brought to the attention of the TA or instructor within one week of you receiving your returned graded report.** Reports turned in after this time are considered late and points will be deducted (Appendix E). Your lab reports count for 50% of your grade so it is essential these be turned in on time in order for you to do well in CHEM 3472.

Grading:

Your course grade will be determined as follows:

Three in-class exams (40% total; Hour exams, 10% each; Final exam, 20%); Homework and quizzes (10%); Lab reports (50%)

Please note:

Lab report grading policy is made by your TA. All discussions regarding grades must take place within one week after your lab is returned to you.

Lectures:

In order for you to conceptionally understand how the instruments work it is important that you attend class and read the assigned material in your text. It is impossible to cover all of the concepts before you begin your experiments. Therefore you may have to read ahead in the text and ask for your instructor's help before beginning an experiments whose concepts have not yet been covered in class. In general lecture notes will not be posted. Therefore it is important that you take notes in class. **All material presented in class is fair game for exams.**

Integrity:

Plagiarism includes copying material from printed sources including the world wide web. To make it your own you must read and understand the material and report the concepts in your

own words. Copying figures from the web is OK as long as it is not covered by copyright and as long as your reference is cited. **Do not give your lab report to anyone, either before or after it is graded.** Should your words appear in another student's report you, along with the student who benefited, will be guilty of collusion. **Those students who either innocently or knowingly participate in collusion or plagiarism will be turned into UTD judicial. Lab reports may be routinely screened with turnitin software. Reports with Turnitin scores of more than 35% will be given a grade of zero.**

Field Trip Policy:

There are no planned field trips in this course.

Comet Creed:

This creed was voted on by the UT Dallas student body in 2014. It is a standard that Comets choose to live by and encourage others to do the same:

"As a Comet, I pledge honesty, integrity, and service in all that I do."

Appendix A: UT Dallas Syllabus Policies and Procedures:

The information contained in the following link constitutes the University's policies and procedures segment of the course syllabus.

Please go to <http://go.utdallas.edu/syllabus-policies> for these policies.

Appendix B

LECTURE, LABORATORY, AND EXAM SCHEDULE

(The dates, order of presentation and topical coverage are subject to change. The correspondence between the material covered and the chapters in the text is approximate.)

| <u>Nominal Date</u> | <u>Lecture Topic</u> | <u>Reference</u> (SH&C or other) |
|---------------------|---|-------------------------------------|
| Tu 08/23/16 | Class Organization, course overview | handout |
| Th 08/25/16 | Organization, Laboratory Reports, Statistics1 | Notes, Harris Ch 2-5 |
| Tu 08/30/16 | Statistics2, S/N, LOD/LOQ | Notes, Harris Ch 2-5 |
| Th 09/01/16 | Statistics3, t-test, ANOVA, LLS | Notes, Harris Ch 2-5 |
| Tu 09/06/16 | Introduction to spectroscopy | SH&C Ch 6 Harris, Ch 18 |
| Th 09/08/16 | Components of optical spectrometer | SH&C, Ch 7 |
| Tu 09/13/16 | Introduction to optical atomic spectroscopy | SH&C, Ch 8 |
| Th 09/15/16 | Atomic Absorption and emission spectroscopy | SH&C, Ch 9 |
| Tu 09/20/16 | Molecular Absorption and Emission spectroscopy | SH&C, Ch 14 |
| Th 09/22/16 | Molecular Absorption and Emission spectroscopy | SH&C, Ch 15 |
| Tu 09/27/16 | Molecular Absorption and Emission spectroscopy | SH&C, Ch 15 |
| Th 10/04/16 | Infrared Absorption & Raman | SH&C Ch 16-18 |
| Tu 10/06/16 | Infrared Absorption & Raman | SH&C Ch 16-18 |
| Th 10/11/16 | 1 st one hour exam review | |
| Tu 10/13/16 | 1 st hour exam | |
| Th 10/18/16 | NMR1 , 1D and 1D FT, components of NMR spectrometer | SH&C, Ch 19 |
| T 10/20/16 | NMR | |
| Th 10/25/16 | Mass Spectrometry | Ch 11A-11B-3 |
| Tu 10/27/16 | Mass Spectrometry | Ch 20A-20C-3 |
| Th 11/01/16 | Introduction to chromatography | SH&C, Ch 26 |

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|----|-------------------|--|-------------|
| Tu | 11/03/16 | GC | SH&C, Ch 27 |
| Th | 11/08/16 | HPLC | SH&C, Ch 28 |
| Tu | 11/10/16 | ICP-MS | Ch 11C-1 |
| Th | 11/15/16 | Review for 2 nd exam | |
| Tu | 11/17/16 | 2 nd one hour exam | |
| Th | 11/29/16 | TEM,SEM | |
| Tu | 12/01/16 | MRI | SH&C, Ch 19 |
| Th | 12/06/16 | Review for Final Exam | |
| F | 12/09/16-12/15/16 | Final Exam, will be scheduled on course lookup | |

Appendix C

LABORATORY SCHEDULE

Students will work in pairs. 10 lab modules will be available; each module requires one week of lab work (two lab periods).

1. Multicomponent UV Analysis of α - and β -Acids in Hops
2. Determination of Mn in vitamin tablet using Atomic Absorbance
3. Determination of quinine in tonic water using fluorescence spectroscopy
4. Use of Fluorescent Plate Reader/Sampling of Heterogeneous Solids
5. Raman Forensics Lab
6. Optimizing Separation Using An HPLC Simulation Routine
7. Using Infrared Spectroscopy to Investigate Protein Structure
8. Analysis of Excedrin Migraine Tablets by Proton NMR
9. Investigation of Imposter Perfumes Using GC–MS
10. ICP-MS analysis of heavy metals in hair samples

Lab/Alternate Activity Schedule

(Assignments for Lab schedule/partners will be made approximately one week prior to the start of labs.)

| Date | Activity | Report Due |
|-------------|----------|---------------|
| T 09/13/16 | Lab #1 | |
| Th 09/15/16 | Lab #1 | |
| T 09/20/16 | Lab #2 | |
| Th 09/22/16 | Lab #2 | |
| F 09/23/16 | | Lab Report #1 |
| T 09/27/16 | Lab #3 | |
| Th 09/29/16 | Lab #3 | |
| F 09/30/16 | | Lab Report #2 |
| T 10/04/16 | Lab #4 | |
| Th 10/06/16 | Lab #4 | |
| F 10/07/16 | | Lab Report #3 |
| T 10/11/16 | Lab #5 | |

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|----|----------|---------|----------------|
| Th | 10/13/16 | Lab #5 | |
| F | 10/14/16 | | Lab Report #4 |
| T | 10/18/16 | Lab #6 | |
| Th | 10/20/16 | Lab #6 | |
| F | 10/21/16 | | Lab Report #5 |
| T | 10/25/16 | Lab #7 | |
| Th | 10/27/16 | Lab #7 | |
| F | 10/28/16 | | Lab Report #6 |
| T | 11/01/16 | Lab #8 | |
| Th | 11/03/16 | Lab #8 | |
| F | 11/04/16 | | Lab Report #7 |
| T | 11/08/16 | Lab #9 | |
| Th | 11/10/16 | Lab #9 | |
| F | 11/11/16 | | Lab Report#8 |
| T | 11/15/16 | Lab #10 | |
| Th | 11/17/16 | Lab #10 | |
| F | 11/18/16 | | Lab Report #9 |
| T | 11/22/16 | no lab | |
| Th | 11/24/16 | no lab | |
| M | 11/29/16 | | Lab Report #10 |
| T | 11/29/16 | no lab | |
| Th | 12/01/16 | no lab | |
| T | 12/06/16 | no lab | |

Appendix D: Chemistry 3472 Lab Expectations

You will be expected to turn in your lab report and copies of lab notebook. You will be expected to show your lab book containing a brief introduction and detailed procedure for your lab each week before you enter the lab. You will not be able to enter the lab until this is complete.

Lab Report

A typed report and an electronic report is expected to be turned in containing the following:

Title, date, partner's name (your name and date on each page)

Abstract

Introduction – This should detail the theory and function of the instrument.

Experimental methods/Procedure - Include serial numbers of the instrument used.

Results and Calculations

Discussion

References

Lab Book page copies attached as an appendix

The lab book should contain an intro, hand written procedure, experimental data, calculations, and brief conclusion. It should also have a complete Table of Contents

You should only write in your lab notebook. **DO NOT** write on loose paper and transfer to your notebook. Your lab notebook should be signed by the TA or the instructor before you leave for the day.

- ❖ You will lose points if you do not clean up. **Before you leave the lab, clean around the balances, under the hood and return chemicals from where you got them.** Make sure to collect all your glassware.

Lab Drawers

Each week you will be expected to come to lab prepared for your specific lab module. You will rotate drawers depending on which lab module you are doing.

Those students doing HPLC-Sim do not have drawers.

Each week when you have completed your lab you will have a TA check to make sure all the glassware is in the drawer before you leave lab. If you fail to check out this could result in charges for missing glassware.

If you finish the lab the first day, you do not have to attend the 2nd lab period as long as you have checked out with a TA before you leave.

Appendix E

LATE REPORTS POLICY

Reports turned in before 8 am on Monday will be 5% off. Those turned in before 8 am Tuesday are 15% off. After 8 am Tuesday they will be 25% off, etc.

All lab reports received after 5 PM will be penalized at a deduction rate of 5%. Each additional business day a lab report is late will be penalized at an additional deduction rate of 10% per day. Lab reports submitted later than 2 weeks after due date equal a zero (0) but we will still give 10 points for the notebook pages.

% maximum credit

| <u>F(due)</u> | <u>S</u> | <u>Su</u> | <u>M</u> | <u>T</u> | <u>W</u> | <u>Th</u> | <u>F</u> |
|---------------|----------|-----------|----------|----------|----------|-----------|----------|
| 100 | 95 | 95 | 95 | 85 | 75 | 65 | 55 |

After one week you will only receive a maximum of 55%. Lab reports turned in two weeks late will receive no credit.

| <u>Second week:</u> | <u>F</u> | <u>M</u> | <u>T</u> | <u>W</u> | <u>Th</u> | <u>F</u> |
|---------------------|----------|----------|----------|----------|-----------|----------|
| | 55 | 50 | 40 | 30 | 20 | 10 |