# Syllabus for CHEM 309

Principles of Instrumental Analysis, Fall 2022, MWF 9:00 AM McGlothlin-Street 20
Thursday 7:00-7:50 PM ISC 1221

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Office Hours: Tuesday (10:00-11:30) and Wednesday (1:30- 3:00)

**Text**: Harris, *Quantitative Chemical Analysis, 9 ed.* - recommended

**Thursday evenings**: from 7:00-7:50 have been set aside for use for CHEM 309L. The first meeting will be for lab. The remaining meeting times will be used for five quests (half-tests) (REQUIRED) (7:00-7:50), and help sessions (optional) (7:00-7:50). These meetings will be in ISC 1221.

## Grading:

| | | |
|---|---|---|
| Quests | (225 points) | 37.5 % |
| Problem Sets | (75 points) | 12.5 % |
| Laboratory | (150 points) | 25 % |
| Final Examination | (150 points) | 25 % |

## Examinations (225 points, 37.5%):

There will be five 50-point quests (quiz/tests). They will be given during the 1-hour block set aside on Thursday evenings (September 22$^{nd}$, October 6$^{th}$, October 20$^{th}$, November 3$^{rd}$, and December 8$^{th}$) from 7–8. These quests will be closed-notes and based on examples given in both the class notes and the text. Your lowest quest grade will only count 50%. Make-up quests will not typically be given. If you must miss a quest, please submit the reason to me in writing one week in advance. If the excuse is valid, the quest may be taken early. If you are sick, I will prorate the missed quest based on your scores from the other quizzes.

**Final Examination (150 points, 25%)** The final is on Wednesday, December 14$^{th}$ at 9:00 AM. The final will be cumulative.

**Homework**: (**75 points, 12.5%)** There will be seven problem sets given out over the semester. These need to be completed using Gradescope. I will post a .pdf template that you can download, fill out, and then upload with your completed work to Gradescope. There will be more than 75 points available across the homework sets, so your final grade will be adjusted by percentage of the total points to 75 total.

**Laboratory (150 points, 25%)** In the lecture portion of the course, you will learn about modern instrumentation, sample prep and data analysis. To truly understand a chemical instrument, it is often necessary to use that instrument in an experiment. To this end, we have designed 8 experiments to illustrate the instrumental techniques that you will learn in the lecture and to give you an opportunity to see the power of modern instrumentation in action. As CHEM 309 is a 4 credit course with integrated laboratory, the lab portion of the course will comprise 25% of your

grade. In order to pass the course, you must complete each of the experiments and turn in the associated lab reports. Lab reports must be turned in by 5:00 PM of your lab section day during the week that they are due.

| Title | points | due date (week of) | grader |
|---|---|---|---|
| I. Gas Chromatography –Whiskey | 20 | 9/26 | McNamara |
| II. GC-MS: haloforms | 20 | 10/3 | Meldrum |
| III. HPLC | 10 | N/A | N/A |
| IV. Tandem Mass Spectrometry | 20 | 10/24 | Poutsma |
| V. LCMS: proteomics. | 15 | 10/31 | Poutsma |
| VI. Atomic Absorption Spectroscopy | 20 | 11/14 | McNamara |
| VII. Fluorescence Spectroscopy | 20 | 11/28 | Abelt |
| VIII. Organic Unknown | 25 | Friday 12/9 | Meldrum |

**Paper assignments (up to 3 points of extra credit towards quests):** During the course of the semester, I will post three papers from the current literature on topics that we are covering in the course: one on separation science, one on mass spectrometry, and one on spectroscopy. If you provide a one-page (single-spaced) summary of the paper, you can earn up to 1 extra-credit point per summary that will be added to one of your quest grades. The summary should be in paragraph format and should include the following:

title of paper and authors with affiliations
statement of the chemical problem that they were addressing
summary of analytical technique used to address the problem
summary of **important** results
summary of conclusions of the paper (did they answer the question or address the problem that they were investigating)
summary of future work motivated
opinion of the paper in terms of style, organization, clarity, significance of the work.

## COVID-related information

Given W&M's policy on COVID, it is expected that this course will be held in person with no remote components. This includes the lab portion of course: CHEM 309L. COVID illness is to be treated as any other illness. I will be teaching this class using PowerPoint slides and will annotate them during the lecture. I will post the PowerPoint slides before lectures and will upload annotated versions after we have completed various sections. I am not planning to record the lectures. Attendance in class is vital to your success in the course. Lab sections will be fully in person and will not be recorded. If you are quarantined and miss a lab, you may make up that lab if the rotation is still going. If the rotation has ended, you will be given data that will allow you to complete the write-up by the assigned due date.

If you are sick, COVID or otherwise, your first obligation is to get better and avoid getting others sick. Do not come to class is you are not feeling well. If you must miss class for any reason, please obtain "notes" from one of your classmates. If you have symptoms related to COVID, get tested and act accordingly.

## Thursday Night Schedule

| | |
|---|---|
| September 1st | Lab intro meeting (REQUIRED) |
| September 8th | Problem set 1 (REQUIRED) |
| 15th | help session |
| 22nd | QUIZ 1 (chromatography) REQUIRED |
| 29th | no meeting |
| October 6th | Quiz 2 (mass spectrometry) REQUIRED |
| 13th | no meeting, Fall Break |
| 20st | Quiz 3 (instrumentation/spec. theory) REQUIRED |
| 27th | no meeting |
| November 3th | Quiz 4 (atom. and molec. spec.) REQUIRED |
| 10th | help session |
| 17th | help session |
| 24th | no meeting Thanksgiving |
| December 1st | help session |
| December 8th | Quiz 5 (IR, NMR) REQUIRED |

# Course Outline

Reading assignments in Harris are indicated in parentheses

I Introduction, statistics, error, [ ] units, measurement, signal to noise, figures of merit (Harris Chapters 3 and 4, and handout on Blackboard)

II. Chromatography theory (Chapter 23)
- A. solvent extraction and chromatographic phases (23.1-23.2)
- B. retention (23.3)
- C. plate theory (23.4)
- D. rate theory (23.5)

III. Chromatography techniques
- A. gas chromatography (Chapter 24.1-24.3)
  1. mobile phases (24.1)
  2. stationary phases (24.1)
  3. sample introduction (24.2)
  4. detectors (24.3)
  5. temperature programing (24.1)
- B. liquid chromatography (Chapter 25)
  1. mobile phases (25.1)
  2. stationary phases (25.1)
  3. sample introduction (25.2)
  4. detectors (25.2)
- C. other LC techniques (Chapter 26.1-26.3)
- D. quantitative and qualitative analysis by chromatography
- E. capillary electrophoresis (Chapter 26.6)

IV. Mass spectrometry (Chapter 22)
- A. general (22.1)
- B. instrumentation
  1. sample intro/ion sources (22.1, 22.3, 22.4, 22.5, 22.6)
  2. mass spectrometers (22.1, 22.3)
  4. detectors (22.3)
- C. interpretation of mass spectra for organic and biological systems (22.2, 21.5)

V. Spectroscopy – theory (Chapter 18)
- A. electromagnetic radiation (18.1)
  1. wavelength/frequency
  2. absorption and emission of radiation
     a. atoms (18.1, 18.6)
     b. molecules (18.1, 18.6)
- B. generalized instrumental design (Chapter 20)
  1. radiation sources (20.1)
     a. continuous sources
     b. line sources
     c. lasers
  2. monochromators (20.2)
     a. prisms
     b. gratings
  3. detectors (20.3)

a. phototubes and photomultiplier tubes
b. diode array detectors
c. thermal detectors

C. Beer's law and deviations (18.4)

VI. Spectroscopy – applications
A. atomic absorption spectroscopy (Chapter 21)
1. theory (21.1)
2. quantitation
3. instrument design (21.2, 21.4)
B. atomic emission spectroscopy (Chapter 21)
1. instrument design (21.2, 21.4)
2. qualitative and quantitative analysis
3. comparison of AAS and AES
C. uv/visible spectroscopy (Chapter 18)
1. theory (18.6)
2. quantitation
3. instrument design (21.2, 21.4)
D. fluorescence and phosphorescence spectroscopy (Chapter 18.6, 18.7)
1. theory
2. quantitation
3. instrument design

VII. Infrared spectroscopy (handouts and course notes)
A. theory (handouts and course notes)
B. qualitative analysis (handouts and course notes)
C. instrumentation (Chapter 20.5)
D. spectral interpretation (handouts and course notes)

VIII. Nuclear magnetic resonance (handouts and course notes)
A. behavior of magnetic nuclei
B. proton NMR
C. carbon NMR
D. instrumentation
E. spectral interpretation

IX. Combined spectral interpretation (material from sections IV, VI, and VII)
A. determining the chemical formula
B. putting all the pieces together

X. Electroanalytical chemistry theory (Chapter 14)*
A. review of basic electrochemistry (14.1)
1. oxidation reduction reactions and half-cell reactions (14.1)
2. units
3. redox potentials
4. free energy, & equilibrium (14.3-14.5)
5. concentration effects – Nernst equation (14.4)
B. galvanic (voltaic) cells (14.2)
C electrolytic cells (14.2)

XI. Electrochemisty Applications*

- A. potentiometric techniques (Chapter 15 and 16))
  1. reference electrodes (15.1)
  2. indicator electrodes (15.2)
  3. pH electrode (15.5)
  4. potentiometric titrations (Chapter 16)
- B. coulometric techniques
  1. constant potential
  2. constant current
  3. electrogravimetry

*Time permitting. This material will not be on any quests but may be on the final exam.