

CHEMISTRY 311  
**INSTRUMENTAL ANALYSIS**

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### **RATIONALE AND OBJECTIVES**

The demands of modern society for materials which are more inexpensive, convenient, nutritious, and/or safe continues to create, in turn, a demand for more analyses of smaller amounts of more exotic substances in less time than ever before. To meet these needs the analytical chemist has turned largely to instrumentation, with the result that the modern analytical laboratory increasingly resembles a cross between an electronics shop and a computer facility, with a seemingly out-of-place article of glassware here and there. Instruments come in a variety of forms to perform a variety of tasks. As with any tool, each performs a particular job well and other jobs not so well, occasionally strikingly so. Any idiot knows that a hammer is useful for driving nails, and a bit of experimentation will reveal that it is virtually indispensable for that purpose. It is hard to imagine, however, how one might efficiently use it to paint a wall.

While it is clearly impossible to cover the entire gamut of modern instrumentation in detail in a single course, several broad categories, incorporating the principles of many common analyses, will be examined. The laboratory will provide an opportunity for hands-on experience with representative instruments, as well as exercise the student's expertise in "doing analytical chemistry".

The goals of this course are several:

- To provide the student with an understanding of the role of instrumentation in the modern analytical chemistry laboratory.
- To familiarize the student with the principles of operation of several representative analytical instruments, thereby fostering an awareness of the strengths and shortcomings of selected instrumental techniques.
- To continue developing in the student an ability to learn to use successfully an unfamiliar instrument or technique.
- To exercise the student's ability to communicate in writing the results of an investigation.

## **COURSE TOPICS**

**ELECTROANALYTICAL TECHNIQUES.** Methods utilizing the electrochemical properties of matter. Potentiometry, coulometry and some common chronoamperometric techniques, including cyclic voltammetry, potential step and complex waveform techniques.

**ANALYTICAL SPECTROMETRIC METHODS.** Methods involving the interaction of electromagnetic radiation with matter. Molecular absorption and fluorescence in the UV and visible regions and atomic emission and absorption using flame and electrothermal atomization.

**CHROMATOGRAPHIC SEPARATIONS.** Theory of chromatographic methods. Optimization of separations. Several currently popular methods, including Liquid-Liquid, Liquid-Solid and Gas-Liquid techniques.

**SPECTROMETRIC IDENTIFICATION OF ORGANIC COMPOUNDS.** modern infrared methods and mass spectrometry. Selected one- and two-dimensional nuclear magnetic resonance techniques.

## **TEXTBOOK**

The following text will be regarded as official for purposes of this course:

Harris, Daniel C.; *Quantitative Chemical Analysis*, Seventh edition (Freeman, 2007) ISBN 0-7167-7041-5

You might recognize this as last term's Quant book. If you own a previous edition of Harris or one of the diminishing handful of texts pitched specifically toward the Instruments course that will do fine too.

## **CLASS WORK**

Class time will be devoted to group-centered guided-inquiry activities designed to encourage the student to build the ability to solve chemistry problems with the help of small-group interaction.. Reading assignments from the textbook will be accompanied by a few questions which the student will be expected to do outside of class.

Active participation on the part of each student is essential to the success of this approach to learning. The contribution of each student has value in the learning process, even though it may not necessarily express "the right answer". Students who withhold their participation are not only refusing to learn themselves, but are also hindering the learning of others. There is therefore a significant contribution to the overall course average for class participation which will be reduced if in the instructor's judgment a student clearly avoids participation on a regular basis.

## **QUIZZES**

From time to time a short impromptu quiz may be administered at the beginning of the class period to encourage students to remain current. They also potentially serve as an indication to the instructor regarding the degree of student understanding without invoking the major contribution to the course average incurred by an examination.

## **HOMEWORK PROBLEMS**

On a regular basis mathematically-oriented problems will be assigned which are to be completed outside of class. These are offered in this way in lieu of including them in class examinations in order to give the student the benefit of having an opportunity to consider the solution over a period of time rather than being expected to come up with it instantly during the exam hour. These problems will be due one week following their distribution. Since these problems function in some ways as examination it is expected that they will represent the student's own best work. The following points are to be strictly observed:

- Students may use any textbook, notes, or other reference material they wish in the preparation of solutions to these problems.
- Students may talk about these problems if they wish with anybody else associated with this course.
- Students may not turn in a solution to any of these problems prepared in whole or in part by somebody else.
- Students may not turn in a solution to any of these problems that has been dictated in whole or in part by anybody else.
- Students must offer in their solutions an organized, legible and sufficiently complete record of the progress from initial data to the final result that partial credit may be unambiguously awarded where appropriate ("show your work!").

## **EXAMINATIONS**

Because the major component of exams, the homework problems, is prepared outside of class, hour examinations will consist primarily of a few short-answer style questions mainly to test the student's understanding of the theory of the course. The student may consult the textbook, his or her own notes, or other references brought to the exam but may not communicate with other students or use materials brought by anybody else.

Examinations are scheduled to be given on the following dates:

First exam	Thursday, September 27
Second exam	Thursday, October 25
Third exam	Tuesday, November 20

A comprehensive final examination will be given as scheduled during the final exam period.

## **CLASS ATTENDANCE**

A formal record of class attendance will not be kept, although the student might bear in mind that in a class of this size it's pretty clear if somebody's missing. Neither is there any direct contribution of attendance to the overall course average. It would be well to note, however, that it is impossible for a student who misses class to contribute to class activity.

Attendance at examinations is mandatory. If the student finds him- or herself, for reasons of illness or other significant inconvenience, unable to appear for an exam, he or she should notify the Dean of Student's office which will circulate a memo to the instructors involved

attesting to these circumstances. Only upon receipt of this memo will a makeup exam be administered. Note that, since it is clearly unfair to the bulk of the class if a makeup is easier than its regular counterpart, and since it is impossible to prepare different examinations of exactly equal difficulty, makeup exams may appear slightly more rigorous than corresponding scheduled examinations. If the student knows in advance that he or she will be unable to appear for an exam as scheduled, it may be advantageous to arrange with the instructor to take it ahead of time.

There will be no makeups of missed quizzes. If a student's absence from class is approved in the manner indicated above, the missed quiz will simply not be counted in the average. Otherwise the score of zero will be assigned that quiz.

## **LABORATORY**

The laboratory component of this course will be divided into two roughly independent parts. We will begin with several quick exercises to familiarize the student with some representative techniques in electrochemistry, spectroscopy and separations, followed by a several week opportunity for each student to pursue a project of his or her own design that makes use of one or more of these techniques. Another small group of exercises related to spectrometric identification of organic compounds will then be offered, followed by an opportunity for the student to identify the components of an unknown mixture.

The following laboratory schedule is anticipated:

29 August – 15 October: familiarization exercises

Ion sensitive electrodes

Potentiometric titration

Chronoamperometry

Formula of a colored complex

Simultaneous UV-vis spectrophotometric determination

Atomic spectroscopy

Gas-liquid chromatography

High pressure liquid chromatography

17 October – 5 November: individual student project

7 – 28 November: familiarization exercises

Quantitative IR

Mass Spectrometry

Nuclear magnetic resonance spectrometry

3 – 10 December: Organic qual mixture unknown

For the familiarization exercises, students will typically work in small groups. Some exercises require one lab period and others two. To complicate matters further, due to the limited equipment available it will be necessary to rotate in order for each group to do each exercise. The schedule has been planned in such a way that groups which rotate on time will be able to complete all of the familiarization exercises without byes, but it will be necessary to stay rigidly on schedule to accomplish this. Groups which miss an exercise or fail to finish it in the time

allotted will typically need to complete the missed work at times other than the scheduled laboratory period.

The lab is not synchronized with the lecture, typically running considerably ahead. The lab handouts will provide the information needed to get results from a given technique but it will probably be helpful for the student to have some familiarity with the handout prior to coming to lab in order to make optimal use of the time allotted.

The group will submit a formal lab report on each exercise not later than one week after the exercise is scheduled to be completed. This bit of timing retentiveness appears necessary because otherwise students tend to do a given exercise as scheduled but fail to do any calculations or other writeup until the last moment, possibly weeks or months later, at which time the significance of the data has been largely forgotten and the benefit of preparing the writeup basically lost. The writeup is to be typewritten and figures, if needed, constructed mechanically or using an appropriate drawing application, much as one might in preparing a manuscript for publication.

Be sure to include the following in your report:

- 1 Unknown numbers where appropriate
- 2 Procedure used, when not prescribed by the lab handout
- 3 Data obtained, as collected
- 4 Graphical or other appropriate representation of data
- 5 Sample calculation
- 6 The results of all calculations
- 7 Comment on significance of result
- 8 Answers to any questions posed in the lab handout

Verbosity is discouraged in favor of a clear presentation of data and results which speaks for itself. The use of clearly annotated tables and graphs, when appropriate, is especially encouraged. When language must be used, a college-level command of edited standard written English is expected (the Bedford Handbook, 7th ed., Hacker, Diana; Bedford/St. Martin's 2006; ISBN 0-312-41933-3 is available in the Bookstore if you need serious help with this). It is probably not practical to attempt to force the above into the format expected of a submission to the chemical literature. You'll be expected to generate one a week anyway and you probably have other things to do, too. Rather, present what needs to be presented in an orderly fashion so that I can see that you did the exercise and understand its significance.

For the project each student will be expected to informally propose an analysis of sufficient complexity to justify three weeks of laboratory time. The writeup for this project will be expected to more closely resemble that expected for publication in a professional journal such as *Analytical Chemistry*.

The contribution of each lab report to the laboratory average will be assigned in direct proportion to the number of laboratory periods allotted for the work represented by that report.

## STUDENT EVALUATION

Much of the work in this course will be done in groups, and artifacts such as group lab reports are appropriately submitted on behalf of the group. Problems, exams and quizzes, on the other hand, are expected to represent the student's own individual best effort, not that of somebody else. Academic dishonesty is grounds for dismissal from the course. You are referred to the document "Academic Honesty at Moravian College" (Moravian College, 1986) for a more complete discussion of this policy.

The final score will be calculated using the following weights:

Class participation	15%
Quizzes	5%
Laboratory	35%
Homework problems	20%
Class exams	10%
Final exam	15%

Letter grades will be assigned in accordance with the following:

final score	grade	final score	grade
92 – 100	A	72 – 77	C
90 – 91	A–	70 – 71	C–
88 – 89	B+	68–69	D+
82 – 87	B	62 – 67	D
80 – 81	B–	60 – 61	D–
78 – 79	C+		

Fractional scores will be rounded to the nearest higher integer prior to assignment of the letter grade.

All material to be considered in the determination of the final grade, with the exception of the final examination, must be submitted on or before Monday, Dec. 10, 2007.