

CHEMISTRY 311  
**INSTRUMENTAL ANALYSIS**

Fall, 2011

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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 chronoamperometry and stripping analysis.

**ANALYTICAL SPECTROPHOTOMETRIC 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*, Eighth edition (Freeman, 2010) ISBN 978-1-4292-1815-3

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 undergraduate Instruments courses, 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 quite beyond "the right answer". Students who withhold their participation are not only potentially 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.

## HOMEWORK PROBLEMS

Many of the class activities include so-called Applications, the ANA-POGIL buzzword describing sometimes divergent problems that require the student to apply the material of the activity to a potentially realistic situation. Assigned Applications are to be addressed by the student outside of class and turned in for a score. Students are permitted to discuss the Applications with one another and may work on them in informal groups of two or more if desired. In the case of a group submission, each member of the group will receive the score assigned the submission.

Homework submissions will be due the class period following completion of the activity of which they are a part. The benefit to the student in addressing these Applications diminishes rapidly as the activity in question fades into antiquity so promptness in submission is essential. Late work will not be scored.

## **EXAMINATIONS**

Three 70-minute in-class examinations will be held. These exams will be administered open-book. The student may consult the textbook, his or her own notes, or any other printed reference brought to the exam but may not communicate with anybody else in any way or use materials brought by others.

Examinations are scheduled to be given on the following dates:

First exam	Thursday, 22 September
Second exam	Thursday, 27 October
Third exam	Tuesday, 22 November

A comprehensive final examination will be given at 1:30 PM on Tuesday, 13 December 2011.

## **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.

Note that this class will not meet on Thursday, 15 September, so that students may attend the Fall Convocation at 10:30 AM on that day.

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.

## **LABORATORY**

With the single exception of the Organic Qual Unknown, students will be expected to perform the laboratory activities in groups. Some exercises require one lab period and others two. To complicate matters further, if there is more than one lab group, due to the limited equipment available it will be necessary for groups 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 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 following laboratory exercises are scheduled:

- Ion Sensitive Electrodes (2 periods)
- Potentiometric Titration (2 periods)
- Chronoamperometry (1 period)
- Anodic Stripping Analysis (1 period)
- Formula of a Colored Complex (1 period)
- Simultaneous UV-vis Spectrophotometric Determination (1 period)
- Molecular Fluorescence (2 periods)
- Flame AA (1 period)
- Graphite Furnace AA (1 period)
- Gas-liquid Chromatography (2 periods)
- High Pressure Liquid Chromatography (2 periods)
- Infrared Spectrometry (2 periods)
- Mass Spectrometry (2 periods)
- Nuclear Magnetic Resonance Spectrometry (2 periods)
- Organic qual mixture unknown (3 periods)

The lab is not synchronized with the lecture in any way. 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 what's in the handout prior to coming to lab in order to make optimal use of the time allotted.

For all exercises except the Organic Qual Unknowns the group will appoint a recorder who will submit his or her activity worksheets on the exercise in question 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 score earned on the submission will be awarded to all members of the group.

Verbosity is discouraged in favor of a clear presentation of data and results which speaks for itself. 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).

A fair amount of work spanning several lab periods is typically in order for the Organic Qual Unknowns near the end of the term. In order to enforce fairness in the distribution of this work each student will be expected to solve his or her own unknowns and report the findings. That report is due on Monday, 5 December 2011.

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. 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	10%
Laboratory	35%
Homework problems	10%
Class exams	30%
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 Wednesday, Dec. 7, 2011.

Students who wish to request accommodations in this class for a disability should contact the Assistant Director of Learning Services for Disability Support, 1307 Main Street (extension 1510). Accommodations cannot be provided until authorization is received from the office of Learning Services.