Course Aims
This is a graduate-level course in which you will be expected to perform with a higher level of insight, independence and sophistication than would be expected in an undergraduate course. There are two very general goals that I want you to achieve in this course:

1. to think like a scientist and
2. to have a familiarity with the fundamental principles and use of modern instrumental methods that will allow you
   a. to solve practical problems using the knowledge gained in this course (which adds on to the knowledge you have acquired as an undergraduate1) and
   b. to use your knowledge and scientific discipline to understand and apply new analytical techniques to meet new experimental challenges (which is really only a very specific application of point #1, thinking like a scientist).

In essence, I would like this course to give you skills so that if one day you are confronted with a difficult analytical challenge you can say “Oh, to solve that problem I need to measure the oxidation state of the surface atoms. I remember that technique X can tell me the oxidation state of bulk atoms—perhaps a variation of that technique (i.e. extension of the fundamental principles) could get me surface information”, then either discover such a variant in the literature or invent it and finally apply it correctly.

Analytical chemistry includes far too many techniques to cover every single one in depth, so the focus of this course is to study a small number of topics in detail with an emphasis on understanding fundamental principles and learning how to extend those principles to other techniques. A survey approach will be used to expose you to a broader range of analytical techniques whose operation can be understood by extension of the fundamental principles covered. Success in the course will depend on the formal approach of the scientific method—to guide your path from observation to conclusion and to guide your presentation of conclusions. You must pay attention to observations and draw conclusions based on those observations in concert with your prior knowledge, and then clearly explain this logic, clearly stating how observation and fundamental principles support your conclusions. You will be more successful in this course, for example, in

---

1 As a graduate student, you should be actively improving your knowledge base and this may required very focused review of undergraduate material so that you can use it as the basis for more easily acquiring new knowledge. If your undergraduate training was deficient in some areas, now is your opportunity to fix it. I am happy to recommend reading and provide guidance, but it is your responsibility to do the work needed.
sitting and thinking about a problem set than in looking for an “accepted answer” somewhere.

Overall, you are expected to work towards improving your ability to conduct scientific research: to understand measurement techniques, design and critique experiments, and to draw conclusions and evaluate the conclusions of others. Important aims include

1. gaining knowledge of physical principles governing instrumental measurements,
2. gaining familiarity with the range of modern analytical tools and the problems to which they apply
3. developing critical, systematic and creative thinking—the scientific method
4. developing the ability to clearly explain yourself within the context of the scientific method. Clear statements of goals, limitations of experiments and the formal and explicit linking of conclusions to their antecedent observations
5. learning how to use an existing, limited knowledge base to acquire new knowledge

The course culminates in a final project in which you . Devotion to learning and adhering to the scientific method and to learning the techniques and principles of instrumental measurements will prepare you for this final project.

Professor
Dr. Jason R. Dwyer
Pastore 318
jdwyer@chm.uri.edu (Please put CHM512 in the subject line)

Lectures
Tuesdays and Thursdays, 9:30-11:00, Pastore 219

Office Hours:
Drop by (Pastore 318/330/332) or by appointment.

Recommended Text
This course will draw on the literature, but Skoog, Holler and Crouch’s Principles of Instrumental Analysis, 6th ed., is strongly recommended. You will find other textbooks, such as standard physical chemistry textbooks, will be useful from time to time.

Course Grading
Classroom preparation, assignments and participation: 30%
Tests (2): 30%
Final project: 40%

Attendance
Attendance in lectures is mandatory. Attendance at several of the chemistry department seminars (Mondays, 3pm, Pastore 234) may be mandatory unless you provide
documentation of a legitimate scheduling conflict. These seminars may form part of the material on which you will be tested.

**Regulations**
Any requirements of this syllabus are in addition to the University of Rhode Island rules governing academic conduct.

**Required Lecture Materials**
1. Scientific calculator. Computers, internet-capable devices and cell phones are prohibited during quizzes, tests and examinations.
2. Pen

**Testing and Assignment Regulations**
1. Any submitted material not in permanent ink cannot be submitted for regrading after it has been returned to the student.
2. You should have a scientific calculator. Computers, internet-capable devices and cell phones are prohibited during quizzes, tests and examinations. Any device enabling wireless communication during a test is prohibited.

In cases of illness only, missed assignments and tests will be rescheduled when feasible, with alternative arrangements made as appropriate. For assignments and tests that cannot be retaken, the final exam will be weighted more heavily to compensate.

**Academic Honesty**
Academic dishonesty in any form is considered a serious offence, and disciplinary action will be taken immediately. The URI policy on academic honesty is detailed in the student handbook (available online), and it is summarized below:

*Students are expected to be honest in all academic work. A student’s name on any written work, including assignments, lab reports, papers, or exams, shall be regarded as assurance that the work is the result of the student’s own thought and study. Work should be stated in the student’s own words, and produced without assistance (or properly attributed to its source). When students are authorized to work jointly, group effort must be indicated on the work submitted.*

The following are examples of academic dishonesty:
- Unauthorized communication during exams.
- Unauthorized use of another’s work or preparing work for another student.
- Taking an exam for another student.
- Altering or attempting to alter grades.
- The use of notes or electronic devices such as calculators, computers, or cell phones to gain an unauthorized advantage during exams.
- Fabricating or falsifying facts, data, or references.
- Facilitating or aiding another’s academic dishonesty.
When there is an allegation of academic dishonesty, the instructor may:
• Fail the student for the assignment, or recommend that the student fail the course.

Students in CHM 512 are encouraged to work in groups on assignments and reports, but the names of all participating students should appear on each submitted assignment and report. If one student in particular is responsible for a key concept in the solution or write-up, then s/he should be given explicit credit right next to that line of the report. Note that all submitted work must still be completed by each student in his or her own words: shared text is not permitted. Simply making small text substitutions (e.g., “But” instead of “However”) or rearranging sentences, for example, are not consistent with the expectation that each student is reporting his or her work in his or her own words. For the final project, however, >90% of the effort, ideas, content, etc. should be directly attributable to the submitting author. You may discuss the final project with others, but the work should clearly be done by the individual submitting author.

Scholarly References
You are expected to use scholarly, peer-reviewed reference sources (e.g., peer-reviewed journals such as Analytical Chemistry, textbooks, handbooks) when citing information. Use of non-scholarly sources such as Wikipedia and “essay stores” in your submitted work will be subject to penalty. You may refer to corporate literature when it is the only source of technical information: the web site and date of access should then be listed in your citation. If you are in doubt, please come and ask me.

Primary citations
Many times “Paper B” will ostensibly report a claim from “Paper A”. If you are using the claim from “Paper A”, you must (1) check that “Paper A” actually makes that claim, and (2) cite “Paper A”, not “Paper B”. Too often “Paper A” doesn’t actually claim what “Paper B” claims it does, or “Paper A” has been misreferenced through the years because people have never actually read it.

Final project
Your final project will be an extensive referee report on a published scientific paper that features chemical instrumentation as a central part of the paper’s results. The referee’s report will summarize the results of the paper and provide an expert evaluation of the experimental design, conclusions and quality of the work. You will also act as the journal editor for a classmate’s referee report, evaluating both the quality of the report (grammar, clear exposition) and scientific validity. You will therefore receive a mark for both your referee report and your editor’s report.

For the referee’s report, you should provide a 1-1.5 page summary of the paper, in your own words. It should show that you read and understand the paper and should be written in abstract form—make it clear and smooth prose with clear logic. Then you will give a scientific evaluation of the paper in its entirety, but making sure to address in extra detail whether the particular analytical technique can actually deliver the results that they claim
it does. You would want to write things like “the authors have not definitively shown that their experiment proves…” or “the authors should perform an additional control experiment in order to…” or “The authors have performed all of the necessary control experiments and the analytical method is capable of deliver better than the spatial resolution they require in order to…” You can write this part as sections with headings or as a combination of paragraphs and some point form sections. I think that you should aim for 3-4 pages (1.5 line spacing), but some papers may need more. So: 1-1.5 pages of the 3-4 is for your abstract, the remainder for your critical evaluation of the paper.

Papers dealing with methods such as the following would be good choices, although I am open to suggestions. All choices must be cleared with me.

Suggested topics: atomic force microscopy, scanning tunnelling microscopy, ellipsometry, second-harmonic generation, XANES/NEXAFS/EXAFS, Förster resonance energy transfer (FRET)-based detection (incorrectly termed “fluorescence resonance energy transfer” in the majority of the literature), MALDI-MS, single-molecule fluorescence.

**Tentative Topics**

Additional topics may be added and/or substituted, as time demands/permits.

(1) Measurement fundamentals:
   a. analog vs. digital signals
   b. data sampling
   c. Nyquist sampling theorem
   d. sensitivity
   e. dynamic range
   f. bandwidth
   g. speed
   h. data conversion
   i. sources of error and compensation
   j. grounding
   k. basic circuit elements: RC circuits
   l. data filtering

(2) Molecular spectroscopy
   a. UV-visible
   b. Infrared spectroscopy
   c. Raman spectroscopy, including surface-enhanced techniques
   d. fluorescence spectroscopy, including FRET

(3) Surface and thin-film analysis
   a. atomic force microscopy (AFM)
   b. scanning tunneling microscopy (STM)
   c. ellipsometry
   d. surface plasmon resonance (SPR)
   e. electron-based techniques
(4) X-ray based methods of analysis
   a. absorption spectroscopy
   b. crystallography

(5) Separation methods
   a. Fundamentals
   b. HPLC
   c. GC
   d. capillary electrophoresis

(6) Microfluidics, lab-on-a-chip devices and nanofabricated devices
   a. Principles & fundamentals
   b. Applications
   c. Characterization
   d. Cytometry
   e. Coulter counting

(7) Pulsed laser methods of analysis
   a. second harmonic generation
   b. sum frequency generation
   c. coherent antistokes Raman spectroscopy (CARS)