Chemistry 122: Advanced Analytical Chemistry

Stephen M. Contakes
Westmont College, Fall 2019 Semester

Lecture & Lab Times:

Lecture: Mondays & Wednesdays, 5:25 – 6:15 PM (can be moved earlier if that would work with everyone's schedule)
Lab: Thursdays, 1:15 – 5:15 PM in Whittier Sciences 202 and 204

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A. Instructor & office hours information:

Instructor:

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Office Hours:

Mondays, Wednesdays, and Friday's 9:30-10:30 AM, Thursdays 10 AM – noon, and by appointment.

B. Course Description (from the University Catalogue):

CHM 122 Advanced Methods in Chemistry (3) Prerequisites: CHM 101 and CHM 121 or consent of instructor. Two lectures and one four-hour laboratory per week. An advanced integrated laboratory course that develops students proficiency with the use of advanced synthetic and instrumental methods and their application to small molecule, metallurgical, polymeric, nanoscale, food, and environmental systems. Laboratory work focus on the selection, design and implementation of instrumental and wet chemical methods and the development of problem solving, troubleshooting, scientific information literacy, and professional communication skills.

C. Instructional goals & objectives:

Student Learning Outcomes (things we will attempt to achieve):

1. You will develop a basic understanding of polymers and how they differ from small molecules.
2. You will learn about the concept of a polymer molecular weight distribution, the different types of molecular weight averages that are used to characterize it, and how those averages correlate with different polymer properties.
3. You will learn about the different classes of polymers, common synthesis techniques used for the production of each, and how the synthesis conditions affect polymer molecular weight distributions.
4. You will gain experience with the synthesis of addition polymers using Schlenk line-based inert-atmosphere synthesis techniques.

5. You will learn about how polymer molecular weight distributions may be determined using gel-permeation chromatography and apply that technique to the analysis of an actual sample.
6. You will learn about nanoscale chemical systems, some methods used in their synthesis, and some of the ways their properties differ from those of small molecules and bulk solids.
7. You will gain experience in the synthesis of surface-modified metal oxide nanoparticles and experimentally demonstrate their utility in water-purification applications.
8. You will develop a basic understanding of the principles of magnetochemistry and its application to the characterization of inorganic complexes using magnetic susceptibility measurements and electron paramagnetic resonance spectroscopy (EPR).
9. You will synthesize transition metal compounds and observe how their spectroscopic properties differ from those of diamagnetic compounds.
10. You will gain practical experience using, troubleshooting, and/or developing methods that involve the following instrumental methods of analysis:

    Spectroscopy:
    Uv-vis spectrophotometry, atomic absorption spectroscopy, FTIR spectrophotometry, ^{1}H NMR spectroscopy, EPR.

    Analytical separations & hyphenated techniques:
    Gas chromatography, high performance liquid chromatography/gel permeation chromatography, column chromatography.

    Mass spectrometry & hyphenated techniques
    Electrospray ionization-mass spectrometry (ESI-MS), GC-mass spectrometry.
11. You will develop proficiency in using statistical and regression methods to analyze and describe experimental data.
12. You gain experience with the use of multivariable regression and spectral simulation for the analysis of complex data sets.
13. You will gain experience with the keeping of complete, accurate, & properly annotated lab records.
14. You will learn how to prepare clear, structured, and well-written scientific reports.
15. You will further develop skill in retrieving, reading, and applying information from the chemical literature to laboratory problems and the preparation of appropriately-referenced scientific reports.
16. You will have thought about how professional chemical practice can impact Christians' moral and spiritual development.

Chemistry department program learning outcomes (how the student learning outcomes connect with what the Chemistry department expects of its majors):

1. **Core Knowledge.** Students will demonstrate a breadth and depth of knowledge in chemistry. This goal is advanced by student learning outcomes 1-10 and 15, which will provide you with practical and theoretical experience in advanced instrumental methods and data analysis techniques, magnetochemistry, macromolecular chemistry, nanochemistry, and the use of the chemical literature. The depth of your understanding of these techniques and their application will be assessed through exams, lab reports and notebook entries. In the latter you will be expected to understand each of these techniques and methods to the point where you are able to craft a skillful and appropriate introduction to each lab's experimental strategy; describe the

experiment itself in an organized and insightful way that outlines the key experimental parameters and give a reader a clear idea of what is important about each experiment; present, describe, evaluate, and summarize your results and their limitations in a professional and insightful manner using text appropriately and judiciously supplemented by clear and graphically appealing schemes, figures, and tables. Your technical mastery of how the equipment works will also be assessed orally at the bench in terms of how well you are able to describe each instrument's operation and the function of various artifacts (sources, detectors, columns, etc.) taken from decommissioned instruments and spare parts reserves. This course will also develop your scientific writing skills beyond that expected in earlier courses.

2. **Experimental Design.** Students will be skilled in working in the laboratory and will be competent in experiment design and problem solving by the time of graduation. This goal will be advanced by student learning outcomes 4-5, 7, and 9-14. In particular, the prelab lecture and discussion activities will provide you experience in evaluating the advantages and disadvantages of various analytical techniques for a given application and in selecting appropriate conditions for a given analysis. Your skills in this area will be reinforced and assessed at the course-concluding problem solving workshop.
3. **Christian Connection.** Our students will be experienced at reconciling Christian and scientific world views. They will have a perspective that integrates their scientific and theological beliefs into a seamless whole. Learning goal 16 will focus on some of the practical theology aspects of this integration. Specifically, you will be encouraged to view their attitudes toward laboratory work and behavior in the lab as an outward expression of your beliefs and to reflect on how you might better align your theological convictions and laboratory practices.

General Education Learning Outcomes how the student learning outcomes connect with the writing and speech intensive general education requirements)

This course fulfils Westmont's in-major writing and speech intensive GE requirements for the chemistry major. To meet these requirements you will be required to write a total of six papers, each of which will be graded based on

(a) The degree to which the content and style of your writing conforms to the style and expectations of good scientific writing.
(b) The clarity, logical consistency, and insightfulness of your prose, specifically.
- Your ability to construct a clear central message that includes purposeful and inviting ideas, insightful arguments and reasons to accept these arguments, relevant and substantive supporting material
- The degree to which that message is organized and artfully presented in ways that makes it easy for a reader to understand, evaluate, and grasp the significance of your work.

To help you improve in your writing you have the opportunity to rewrite your first paper and turn it in to be regraded for additional credit. Further guidance on these papers will be provided in the form of written and verbal feedback on your papers and through dedicated class sessions on writing instruction.

Course Methodology (things we will do):

1. Lectures, readings, exercises, and 30-minute exams will be used to develop and assess your understanding of the chemical principles governing the synthesis and behavior of polymers, nanoscale systems, and paramagnetic compounds.
2. You will gain practical experiences with common laboratory instrumentation and techniques through a series of laboratory exercises.

3. Readings and pre-lab lectures will be used to give you an overview of the instruments and techniques used in the various experiments and to prepare you to develop experimental protocols for addressing the lab exercises.
4. Lab notebook entries will be reviewed weekly for quality and completeness.
5. A short tutorial, reading, and set of guidelines will help prepare you to craft well-written lab reports.
6. Written laboratory reports will be used to assess your mastery of laboratory practices, data analysis, and scientific literature and writing skills.
7. A reading assignment and discussion will be used to explore how the practice of chemical analysis can impact our Christian lives.

E. Textbooks and Other Materials:

Required Textbook:

Most readings will involve web-based sources, handouts, and the chemical literature. However, you will also need:

- Walton, David; Lorimer, Phillip *Polymer Chemistry* Oxford University Press, 2001. ISBN-978-0198503897.
- Lab notebook (a composition book is OK as long as the binding is sewn or glued and the pages not perforated)

In addition one reading assignment will require you to access materials through the Westmont library reserve system:

- Hearn, Walter "Whole People and Half-truths" in *The Scientist and Ethical Decision*, Charles Hatfield, ed. InterVarsity Press, 1973. *(the book is on reserve in the library)*
- Saks, M.J., Koehler, J. J. *Science*, **2005**, *309*, 892-895. *(this article is available electronically via the Westmont library)*.

Course Webpage:

The course Eureka page contains a copy of the syllabus and schedule, experiment handouts, and copies of the Lecture PowerPoints.

F. Method of Grading:

The evaluation segments are weighted as follows:

Exams	200 points	20 % total
Formal lab reports	600 points, 100 each, 1 drop	60 % total
First lab report, first draft methods & results	10 points	1 % total
First lab report, partial first draft	40 points	4 % total
Lab Notebook	110 points total, 10 each	11 % total
One page reading assignment summary	20 points	2 % total
Instrumental problem solving workshop	20 points	2 % total
Total	1000 points	100 %

Grading rubrics for the notebooks and formal lab reports are given as an addendum at the end of this
Based on the collected score, letter grades are assigned, where A equals an exceptional, B a superior, C an average and D a poor result. I reserve the right to curve the final grades and grades for individual assignments but will guarantee the following letter grades or better if you achieve the following pre-curve point scores:

A	92.5-100%
A-	90.0-92.4%
B+	87.5-89.9%
B	82.5-87.4%
B-	80.0-82.4%
C+	77.5-79.9%
C	72.5-77.4%
C-	70.0.5-72.4%
D+	67.5-69.9%
D	62.5-67.4%
D-	60.0-62.5%
F	≤59.9%

Incomplete grades will only be considered for medical reasons or significant personal reasons (i.e. death in the family, etc.). Which personal reasons are significant will be determined by the instructor.

G. Course Policies

General Conduct:
As far as possibly, I expect you to conduct yourselves with courtesy, dignity, honesty, and discretion as befitting Christian men and women. This includes but is not limited to honesty in preparation of lab reports, prompt attendance at lectures and laboratories, and diligence in attending to the laboratory safety rules.

Attendance:
Attendance at the weekly labs is expected and mandatory. A missed lab will result in a zero for that assignment. Before leaving lab, students should check out with the instructor. This involves verifying that their lab area is clean and presenting their lab notebook to the instructor for grading prior to leaving lab.

Laboratory Safety Rules:
All students must complete the departmental safety handout/checklist sheet and agree to abide by the departmental safety expectations. A few key points are given below just to emphasize that they are particularly important:

1) All laboratory experiments will be done during the scheduled laboratory periods. The instructor or a teaching assistant must be present during the time work is being done.
2) Safety glasses or goggles must be worn at all times in the laboratory.
3) Gloves should be used for all procedures in which chemicals are handled.
4) *No shorts or open-toed shoes* are allowed in the laboratory.
5) Eating and drinking are not allowed in the laboratory.
6) Do not do anything you know or think might be unsafe. If you are unsure about the safety of any procedure, stop and ask for guidance before proceeding.
7) Clean up spills (especially of acids and bases) as soon as you can and let others know they are there so they can avoid them. Dispose of broken glassware in glass containers.
8) Dispose of used materials in appropriate containers, not down the drain, unless directed otherwise.
9) Clean up your lab area before leaving for the day. Leave it as you found it.

Lab Reports:
Formal reports (FRs) for each laboratory are due at the end of the next class session following the lab. **Reports should be submitted both in hardcopy and as pdf files** so that the instructor can annotate them electronically (*e.g.* legibly).

When preparing your reports you are encouraged to work with other students to discuss the experiments and data analysis. However, all submitted written work must be your own (see the academic dishonesty policy below for more details). Linear regression, standard deviation, and graphing techniques will not be directly taught in this course since these are covered in other lower division chemistry courses. However, since you are expected to use them in your data analyses as appropriate I would be pleased to provide you with one-on-one instruction in any of these techniques. Just let me know and I'd be happy to arrange a time when we can meet for this purpose.

In the instructor's experience science writing is best learned in a coaching relationship. Because of this I highly encourage you to meet with me one or more times throughout the semester to discuss particular graded lab reports or lab report drafts. I would even be glad to grade one or two of your lab reports in your presence and explain the reasoning behind my comments and grading procedures. In either case, please plan at least 30-45 minutes for these meetings. They might take considerable time but in my experience they are one of the most effective routes to improvement.

Late Lab reports will incur a 25% per day grade reduction. Since this is generally more than you are likely to lose due to a complete but imperfect lab report you are highly encouraged to submit even noticeably imperfect reports on time as long as they are complete and you are reasonably sure your data analysis is correct.

Lab report rewrites:
A significant part of this course involves learning how to think deeply about laboratory science and communicate it effectively and professionally. Doing this takes practice and is best learned in the context of a coaching relationship with one or more professional scientists. Not infrequently, failure is the royal road to success. You might even find one or more of your "best effort" lab reports returned with many critical comments and a less than stellar grade. If this happens to you have no fear. This class provides a reasonable safety net to allow you room to learn from your mistakes without undue harm to your final grade. Not only will lab reports be returned one week before the next one is due; you'll have two opportunities to rewrite lab reports. First, you are required to revise one of your first two lab reports based on feedback you receive on your initial drafts. In that case the rewrite will be worth the points of a full lab report and the initial draft much less (30% of what a normal report contributes to your overall grade). Second, you will have the opportunity to rewrite any one lab report for a regrade; in that case the average of the rewrite and original lab report grades will replace the original grade you received. You may do this for any lab, including the one your originally rewrote.

Electronic equipment (cell phones, laptops, calculators, etc...):
Electronic equipment may be freely used during lecture and labs as long as they do not distract the attention of other students. All lab records, aside from instrument data graphs and reports, must be recorded in the laboratory notebook. As far as possible, please leave the room to conduct or receive phone calls.

Academic Dishonesty:
Evidence of academic dishonesty, including plagiarism and falsification of data, will result in a zero for the particular assignment. A second offence will result in withdrawal from the course with a grade of F. As per Westmont college policy all instances of academic dishonesty will be reported to the provost's office for possible further action at their discretion.

Note that plagiarism covers more than verbatim copying of material; it can include copying another student's flow of thoughts and ideas. Consequently those of you who work with other students when writing your lab reports are particularly invited to read Westmont College's plagiarism policy for guidance (http://www.westmont.edu/_offices/provost/plagiarism/plagiarism_policy.html) and to talk with the instructor about any doubtful cases that might arise.

Disability/Medical:

Exceptions to the course policies will only be considered for disability reasons or serious sudden medical or personal emergencies. Students who have been diagnosed with a disability (learning, physical or psychological) are strongly encouraged to contact the Disability Services office (Please contact the Director of Disability Services, Sheri Noble (snoble@westmont.edu) as early as possible to discuss appropriate accommodations for this course. Formal accommodations will only be granted for students whose disabilities have been verified by the Disability Services office. These accommodations may be necessary to ensure your full participation and the successful completion of this course. Serious sudden emergencies must be documented and discussed with the instructor within one week of the student's return to campus.

H. Disclaimer:

The course schedule, topics, assignments, tests, and lab exercises presented in this syllabus are offered in good faith. However, these may be changed at the instructor's discretion with the caveat that additional assignments, tests, and quizzes will not be capriciously imposed on the students.

1) This syllabus and lab report guidelines are adapted and expanded from those written by M. Masuno (2007), N. Tro (2006), and M. Nishimura (1995).

I. Tentative Course Schedule

Since this course emphasizes problem solving and experimental design it does not have a traditional schedule. Instead, you'll just be given a series of problems to solve which you'll attempt one after the other. In some cases a problem will take you only one week to complete while in others it may take several weeks to obtain sufficiently trustworthy results. At various points throughout these exercises you will be given laboratory handouts to use as a starting point for your laboratory work; at other times we will work together as a class to develop our own plan for moving forward. The problems are:

1. Do Sunkist and Mountain Dew contain more caffeine and benzoic acid than the FDA permits?
2. Writing professional quality articles – a lecture, discussion, tips, and tricks.
3. Can we remove heavy metal pollutants from water using surface-modified nanoparticles?
4. Why did a steel beam fail?
5. How effective is a new xylene isomerization catalyst at producing *p*-xylene from petroleum-derived mixtures?
6. How do synthesis conditions affect the molecular weight distribution and viscosity of polystyrene?
7. How does the composition of Laponite:Polyacrylamide Composite Hydrogels affect their Elasticity (Young's Modulus) & Tensile Strength (Break strength)?
8. How far can an understanding of General, Organic, and Analytical chemistry help us make, structurally characterize, and understand the properties of transition metal complexes?
9. (time permitting, unlikely) What complexes form between Ni^{2+} and ethylenediamine in aqueous solution?

Right now the plan is to work through these projects in order, although depending on our progress the schedule may be rearranged.

Final Exam period Chemical Analysis Problem-Solving workshop activity
Friday, Dec. 14th, 8 – 10 AM: Chemical analysis problems workshop

Optional assignment: I will accept a rewrite of any one of your lab reports (including the one you rewrote before). In this case the rewrite grade will be averaged with the original lab report grade. You may complete this at any time but I will not accept it after the Thursday final exam period.

2019 Advanced Analytical Chemistry Expected Schedule

(assumes a M or W and F lecture schedule along with a Th lab schedule; Note that to protect the student experience this schedule is provided for program review use only)

Week	Activities
August	
Mon 26	Course orientation & general principles of scientific writing
Wed 28	More on scientific writing - How to write methods and results sections and Introduction to the first challenge: **Do Sunkist and Mountain Dew contain more caffeine and benzoic acid than the FDA permits?**
Thu 29	Experimental spectrophotometric analysis of soft-drinks; preliminary data analysis; let students know that they will need to write up their work by the middle of next week
September	
Mon 2	More on writing scientific papers - the discussion, conclusion, and introduction sections and the abstract; ask students to write • Results, discussion, and conclusions sections for Wednesday's class • a full paper draft for Thursday's lab
Wed 4	Spectrophotometric analysis of a soft drink results debrief & development of a multicomponent mixture analysis procedure **Partial report (results, discussion, and conclusions) sections for the soft drink analysis lab is due**
Thu 5	Measurement of Beer-Lambert curves for the multicomponent mixture analysis; conduct analysis and plot results **1a. First partial draft of the soft drink analysis lab is due**
Mon 6	Multicomponent mixture analysis data evaluation debrief & introduction of the GC-MS-based $^{13}C_3$-labelled internal standard method
Wed 11	Writing scientific papers suggestions - writing abstracts and the discussion, conclusion, and introduction sections
Thu 12	Soft drink analysis by the GC-MS-based $^{13}C_3$-labelled internal standard method; **introduce the Can we remove heavy metal pollutants from water using surface-modified nanoparticles? problem;** introduce the synthesis and properties of surface-modified nanoparticles; and get students started on the nanoparticle synthesis
Mon 16	GC-MS data debrief; ask students to write up the soft drink analysis paper; and Lecture: What is nanoscience and how do nanoscale chemical systems differ from small molecule and bulk solid systems?
Wed 18	Get students started with drying their nanoparticle samples; lecture: Nanoparticle basics: Types, preparation, and characterization methods
Thu 19	Experimental assay of heavy metal removal by humic acid-modified nanoparticles using AAS **1b. Complete and revised spectrophotometric analysis of caffeine and benzoic acid in soft drinks report is due**
Mon 23	More nanoparticle synthesis, characterization, and properties
Wed 25	Heavy metal removal data evaluation debrief; review of calibration curves, detection, and quantitation limits; Discussion: How might the analysis of metals in water by AAS be improved?

Thu 26	**Introduction to the Why did the Steel beam fail? problem**; start the Mn in steel sample preparation; and complete nanoparticle characterization work
Mon 30	**Exam 1: Nanoscience basics (50 points)**
October:	
Wed 2	Scientific writing debrief & lessons for improvement using *The Science of Scientific Writing* (Lessons 1 & 2 each unit of discourse should have one and only one point; pay attention to how your discourse is flowing by appropriately crafting the topic and stress positions of sentences, paragraphs, and sections)
Thu 3	Analysis of Mn in steel as MnO_4^- using UV-vis, data workup, and meta-analysis of the data **2. Article for the nanoparticle water purification is due**
Mon 7	**Fall Holiday**
Wed 9	AAS analysis of Mn in steel debrief; discussion of error propagation in standard addition-based analysis methods; more *The Science of Scientific Writing* tips (Lessons 3 & 4 – strive for stress-topic agreement throughout your paper and work to avoid logical gaps)
Thu 10	Introduction to the **How effective is the xylene isomerization catalyst at producing *p*-xylene**? problem and discussion of potential analytical approaches, concluding by letting students know we will use proton NMR; Discussion of the instrumental aspects of NMR and how NMR measurements are conducted using modern High-Field NMR instruments; students are checked out as users of Westmont's AAvance 400 MHz NMR; preparation and NMR analysis of BTX mixture samples **3. Article for the Mn in steel analysis is due**
Mon 14	Tutorial: Analyzing and reporting NMR data using Topspin; Xylene isomerization catalyst results debrief
Wed 16	Introduction to polymers, their synthesis, and this week's problem: **How do synthesis conditions affect the molecular weight distribution of polystyrene?**
Thu 17	Introduce radical-initiated addition polymerization and Schlenk techniques for conducting air-sensitive syntheses; Experimental synthesis of polystyrene
Mon 21	Lecture: Polymer synthesis methods and the control of molecular weight
Wed 23	Lecture: The characterization of polymer molecular weight distributions using gel-permeation chromatography
Thu 24	Lab: Analysis of polystyrene samples using gel permeation chromatography **4. The article for How effective is the xylene isomerization catalyst at producing *p*-xylene? Is due**
Mon 28	Tutorial: Calculating polymer MW distributions and average MW values from GPC data and Polystyrene MW distribution data debrief
Wed 30	Lecture: Polymer crosslinking and the mechanical properties of polymers
Thu 31	Introduction to **How does the composition of Laponite:Polyacrylamide Composite Hydrogels affect the Gels' Elasticity (Young's Modulus) & Tensile Strength (Break strength)?** and preparation of Laponite: pomyacrylamide composite rods
Mon 4	Discussion: How to get up to speed on the literature in preparation for starting a new project or writing a paper Tutorial: Using reference retrieval and management software with

Wed 6	Introduction to the **How far can an understanding of General, Organic, and Analytical chemistry help us make, structurally characterize, and understand the properties of transition metal complexes? problem.**
Nov. 7	Measurement of hydrogel stress-strain curves and begin the synthesis of various metal acac complexes. **5. Article for the polystyrene MW distribution lab is due**
Mon 11	**Exam 2: Polymers (100 points)**
Wed 13	Discussion: How might the IR, NMR, and mass spectral techniques you learned about in organic chemistry be applied to the characterization of metal complexes?
Thu 14	Recrystallization and drying of the TM complexes; begin their structural analysis using ^{1}H NMR, ESI-MS, and IR spectroscopy; (time permitting) begin Evan's method data collection.
Mon 18	Metal complex synthesis debrief and lecture: magnetochemistry basics and Evan's method for measuring magnetic susceptibilities: **6. Article for the laponite:hydrogel composite lab is due**
Wed 20	Thanksgiving Holiday – No lab
Nov 21	Thanksgiving Holiday – No lab
Mon 25	Labwork: Prepare Evan's method samples and continue with ^{1}H NMR, IR, ESI-MS, and Evan's method characterization of the metal complexes
Wed 27	Structural characterization of TM complexes debrief and lecture: EPR and its utility for characterizing paramagnetic TM complexes
Fri 28	Lab: Magnetic susceptibility by Evan's method and EPR data collection; inform students that the TM complex lab report will take the place of the final so students should start on it early.
December	
Mon 2	Magnetic susceptibility data debrief; crystal field theory as an explanatory filter
Wed 4	**Exam/Quiz 3: Magnetochemistry (50 points)**
Thu 5	EPR measurements debrief and, if needed, simulate EPR spectra; then discuss the reading reflection papers, complete the course evaluations, and clean up the lab as needed **The reading reflection paper is due**

Final Exam period Chemical Analysis Problem-Solving workshop activity
Friday, Dec. 14th, 8 – 10 AM: Chemical analysis problems workshop
7. Article for the TM complex synthesis & analysis lab is due

Optional assignment: I will accept a rewrite of any one of your lab reports (including the one you rewrote before). In this case the rewrite grade will be averaged with the original lab report grade. You may complete this at any time but I will not accept it after the Thursday final exam period.