

# The University of Vermont

## CHEM 201 Advanced Chemistry Laboratory Chem 201 Syllabus - Spring 2012

### Course Description:

What the UVM Catalogue says about CHEM 201 - Advanced Chemistry Laboratory:

"Laboratory problems requiring modern analytical, physical, and inorganic synthetic techniques."

What we think you are saying at this point in your college career:

I have finished  $2\frac{1}{2}$  years of chemistry. I have completed organic chemistry, and organic lab was awesome. How do I get exciting labs in other areas of chemistry? I mean, freshman chemistry was ok, but lab titrations with a burette are way behind me. I would like to get hands-on experience with modern sophisticated instrumentation and to perform experiments that really challenge my thinking.

Yes boys and girls, that is what we hope we have for you in this two course sequence, CHEM 201 & 202.

CHEM 201 is also a spectroscopy course where you get to learn all about and use:

- Infrared spectroscopy in *lab 1* and again in *lab 2 & 4*
- NMR spectroscopy in *lab 3* and again in *lab 4*
- UV/visible and excitation/emission (fluorimetry) spectrometry in *lab 2*
- Inductively coupled plasma (ICP) atomic emission spectroscopy in *lab 5*
- Mass spectrometry in *lab 6*

### Learning Goals:

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- To apply knowledge of chemical and physical principles to the solution of qualitative and quantitative chemical problems
- To understand the interplay of observational data, hypotheses, and hypothesis-driven experimentation through application of the scientific method
- To become proficient in chemical laboratory techniques and apply these techniques to practical and current problems in research
- To be able to read and critically evaluate the chemical and scientific literature
- To learn to present scientific data clearly and effectively through both written and verbal communication

### Prerequisites that are important:

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- **CHEM 161 (recommended, but not required)** – Because CHEM 201 is the place where we will put in practice the knowledge in quantum chemistry, CHEM 161 is recommended to have been completed prior to CHEM 201. However, the basic knowledge required from CHEM 161 will be repeated in CHEM 201 lectures, such that with some additional reading, a student can complete CHEM 201 without completing CHEM 161 first.
- **CHEM 142 or 144** – means you have finished the 1st 2-years of chemistry. Note: it is possible to take CHEM 201 concurrently with CHEM 144, but it is not advised.
- **CHEM 221** or concurrent enrollment – CHEM 221 is not really a prerequisite for CHEM 201, but what a waste of your time to play with instruments in CHEM 201 and not know how they work! You will learn all about instrumentation in CHEM 221 and get to play with these instruments in CHEM 201. **What a combo: CHEM 221 + 201.**

### Lecture:

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Mon., Wed., & Fri., 1:55-2:45 pm, Angell B203

The foci of Advanced Chem Lab are the laboratory experiments and the lab write-ups and presentation. The purpose of the class time is to provide lectures about the chemistry and instrumentation that will be in each laboratory. As such, we won't need to meet for 3 class days a week.

*See calendar of classes* for which days class will meet.

### Laboratory:

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Thurs., 1:00-7:00pm, Cook A105 & the 2nd floor NMR/GCMS room

### Text:

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No text covers the diverse laboratories in the course. Readings will be provided as (i) photocopies of articles from the scientific literature, (ii) chapters from books and (iii) books on reserve in the chemistry library. Students will also have to search the literature for additional articles and book chapters relevant to particular laboratories.

### Course Instructor:

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**Dwight E. Matthews** 656-8114 Cook A205

## Graduate Teaching Assistants:

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**Daniel DePuccio** 656-4394 Cook A135 - Expts 1, 2, & 5

**Neil Mucha** 656-0932 Cook A107 - Expts 3, 4, & 6

## Office hours:

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Scheduled per student need

## UVM Policy on Absences:

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Religious Holidays: Students have the right to practice the religion of their choice. Students should submit in writing by the end of the 2nd full week of classes their documented religious holiday schedule for the semester if there are any conflicts with the class or laboratory schedule.

*Inter-collegiate Athletics:* Members of UVM varsity and junior varsity teams are responsible for documenting in writing any conflicts between their planned athletic schedule and the class (& laboratory) schedule by the end of the 2nd full week of classes.

## UVM Policy on Academic Integrity:

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Offenses against the Code of Academic Integrity are deemed serious and insult the integrity of the entire academic community. Any suspected violations of the code are taken very seriously and will be forwarded to the Center for Student Ethics & Standards for further investigation. [Details](#)

## How the course grade is determined:

	Points	
Written lab reports (5 x50 points each - see <a href="#">link on lab reports</a> )	250	72%
Oral lab report (1 x50)	50	14%
Lab performance and lab notebook	30	
Participation during oral presentations	<u>20</u>	<u>50</u> 14%
	<b>Total:</b>	<b>350 100%</b>
<b>Optional, bonus lab:</b> Design and complete an additional lab experiment of your own — available <i>ONLY</i> if there is sufficient open lab time.	50	
<b>Failure to submit a lab report:</b> any missing lab report will be assigned a negative grade	-20	

**Update the literature used for the various labs.** Bring me (DEM) a good article from the literature or chapter from a book reviewing or discussing a key aspect of any of the labs *that is newer and better than the existing references already cited for the lab*, and I will give you 5 points up to a maximum of 30 points.

## More Information About Chem 201:

### Lab safety:

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- **You will continue the same laboratory safety practices in Chem 201 as per your earlier labs at UVM in the Dept. of Chemistry.**
- In addition, you will be responsible for looking to see what chemicals and what procedures are being used *in advance* of each laboratory. You will be expected to come to the laboratory already equipped with the necessary safety information concerning chemicals to be used in that laboratory.
- You can get the necessary safety information from [the Chem. Dept Lab Safety page](#) including:
  - [Material Safety Data \(MSDS\) Info.](#)
  - [NIOSH/WHO International Chemical Safety Cards](#)
  - [The UVM Chem Dept Chemistry Safety Bulletin](#) (PDF file)

### Lab notebooks:

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**Yes you need to keep a laboratory notebook.** The notebook is worth up to 9% of your grade. The usual is expected with regard to the notebook:

- The notebooks will be clearly marked with your name and address.
- Each day's entry will be preceded by an entry of the **date** of work.
- All entries will be in permanent ink.
- All entries will be readily readable! Points will be deducted for bad hand-writing and ambiguous numbers.
- All data that needs to be written down (a weight from a balance), will be entered at the time you collect it, not on scraps of paper.
- In this lab, many of the experiments will generate complex data sets using a PC controlling the instrument. You can't write this material down. It needs to be collected in a separate folder or 3-ring binder when it gets printed. You may want to do a "reducing" photocopy of critical pieces of information that need to go in your notebook and use a glue stick to affix them into your notebook. You may also glue typed-procedures into the notebook in place of rewriting

procedures, and you may make references to specific instrument operating instructions etc. in a specific manual or hand-out instead of recording this information directly into your notebook.

- It's helpful if you bring a "thumb-drive" or USB memory stick to lab to download data into a format that can be transferred to Excel for further processing and plotting for lab reports.
- Although much of the data you collect will come from a computer controlling an instrument, you still need to record all of the operating parameters of an instrument at the time you are using it.
- You should organize for every experiment in advance, prior to starting it, and try to arrange space in your notebook for that day's experiment accordingly.
- You need your notebook with you at in the laboratory always.

## Lab reports:

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### Get info

## Lab oral presentation:

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- **Each student chooses one of the six laboratory experiments to present orally to the class, rather than as a written lab report.** The basic format will be the same as the written lab reports. *Communication skills are critical to success in a career in science. Everyone has to present their work during their careers. Here is a perfect opportunity for you to begin in a less threatening environment.*
- You will have ~30 min to present your work and 10 min for the audience (your class & instructors) to ask questions. An overhead computer-projection device projector that connects to a PC and an overhead display camera will be available for your talk, or you may bring your own laptop to hook up.
- The schedule for the oral presentation will be 1st-come 1st-served. You need to send an e-mail to me ([DEM](#)) stating which presentation date you wish to reserve and which laboratory you wish to present.
- [See the link in the menu above](#) for details about the oral presentation.

## General lab format:

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- At the start of each lab, your TA will get together with you to discuss the experiment to be done. This is your best chance to ask questions. Preliminary reading assignments, the general plan for the experiment, etc. will have been given to you at least a week in advance of the experiment.
- Key to success in the course is a positive mental attitude when you arrive to start a lab. We will have done the best we can in preparing an experiment, but Murphy's law will sometimes strike, causing delays or slowing completion of experiments. Should extraordinary difficulties occur, your TA will work with you to extend working hours or to complete the experiment at another time.
- We have planned each laboratory so that all necessary data can be obtained well within the scheduled laboratory time. Although you should plan on being present for the full-scheduled time, we hope to complete the experimental part of each lab in much less than the 6 hours of allotted time.

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## Brief Synopsis of the Laboratory Experiments:

### Lab #1: FT-IR

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**1 lab day.** You will learn how to use a high resolution Fourier transform-infrared spectrometer (FT-IR). We will take advantage of the FT-IR's high spectral resolution to measure various physical chemical vibrational properties of diatomic gases. The 1st gas to be studied will be CO at normal and high resolution modes. The 2nd gas will be HCl. Here you will be able to distinguish the isotopes of  $^{35}\text{Cl}$  and  $^{37}\text{Cl}$ . You will also prepare  $^2\text{HCl}$  and measure the spectrum of  $^2\text{HCl}$ . You will calculate the physical chemistry rotational and rotational-vibrational parameters for both  $^1\text{HCl}$  and  $^2\text{HCl}$ . You will also calculate bond strengths from the measured mechanic parameters of these oscillators.

### Lab #2: Molecular Spectroscopy

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**1 lab day.** You will use our research-grade UV-visible spectrophotometer and our fluorometer to investigate the fluorescence emission spectra of anthracene. The vibronic features of the fluorescence will be related to conventional IR absorptions.

### Lab #3: NMR

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**1 lab day.** You will use the *Varian 500 MHz NMR*. We will take advantage of the instrument's resolution and sensitivity to acquire  $^1\text{H}$  data for the keto-enol tautomerization equilibrium of ethyl acetoacetate and acetylacetone and measure the equilibrium constants in two different solvents.

### Lab #4: Vacuum Chemistry

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**1 lab day.** You will learn how to manipulate, move, sample and measure gases using a vacuum line. Two gases,  $\text{BF}_3$  and  $(\text{CH}_3)_3\text{N}$  will be combined to form a solid-state product. The product will be characterized to confirm the reaction.

### Lab #5: Atomic Emission Spectroscopy by ICP

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**2 lab days.** You will learn to use the *Perkin-Elmer Optima 7000DV* inductively coupled plasma (ICP) optical emission

spectrophotometer to measure calcium, sodium and other ions in ground water by atomic emission. This lab will focus on methods of quantification and interferences.

## Lab #6: GCMS

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**2 lab days.** You will learn modern separation techniques via gas chromatography (GC) and analysis techniques via mass spectrometry (MS). You will use the *Varian Saturn 2100T ion trap GCMS* to make your the measurements. You will also learn chemistry required to prepare biological samples for GC measurement by performing micro-chemistry reactions on samples of amino acids. You will separate the individual amino acids by GC and collect spectra by MS for identification of each compound. Using standard mass spectra analysis techniques, you will confirm the identity of each amino acid peak.



Last modified January 16 2012 05:00 PM

# The University of Vermont

## CHEM 201 Advanced Chemistry Laboratory

### Chem 201 Lab Reports: What is required

The five written lab reports comprise 75% of your grade. Thus, the preparation of the lab reports is expected to take a significant amount of your time spent in the course. For the wise student, much of the time spent should be spent *prior to the lab*:

1. Understanding the goals of the lab and what will happen in the lab before you get there
2. Learning the underlying chemistry to be done in the lab
3. Learning the principles of the instrumentation to be used
4. Thinking about what data sets are to be acquired
5. Thinking about what calculations will be performed
6. And thinking about what interpretations can be made from the calculations.

Your written reports should reflect mastery of these points for each lab.

Each formal, written lab report is due 2 weeks after the lab is scheduled to have been completed. Submit your reports directly to the TA in charge of the lab.

#### The Good News and the Bad News:

- The **bad news**: you lose 1 point (2% of your grade) for each weekday-day that you are late, where 10% represents a change in letter grade.
- The **good news**: you receive 1 bonus point for each weekday-day that you hand in your lab report *ahead of the due date*.

The format we use for the written reports is as close as we can make it to the format that you would use if you were submitting a manuscript for publication to a journal. The skills you will develop in writing these reports will carry over to *anything* else you do in science beyond this course. Good writing skills are as important to the successful scientist as good bench-skills--perhaps more important because if you cannot communicate effectively the results of your work, they will die of ennui in the back drawer of a filing cabinet or in the middle of a dusty 3rd-class journal on a library shelf.



[PDF of the instructions](#) for preparing your lab reports

Yes, it is confusing to organize and write a lab report in manuscript format for the first time. *The problem is organization*: getting your thoughts organized, getting your data organized, & getting your words in an organized fashion. Probably the most difficult part is figuring out which of all the data you collected in a lab should be included in Tables and Figures and which are supplementary and should only be included as Appendix material. That's where the organization part comes in. The only way to get organized is by trying. It will get easier as you write up successive laboratories.

I have also included two files here for you to download as an aid to help you understand what a manuscript in manuscript form looks like when converted to a published paper in a journal. The [1st PDF file](#) is a manuscript submitted to *Analytical Biochemistry* for the purpose of describing a new method to measure glucose and glycerol stable isotopically labeled tracers by liquid chromatography-mass spectrometry. The [2nd PDF file](#) is the paper as it was published from this manuscript in *Analytical Biochemistry*.

- [PDF manuscript](#)
- [PDF published paper](#)

#### Other resources that also have *How to Write Good information*:

- [Plain Language.gov](#) — a web site devoted to assist people in effectively communicating with their audience.
  - This site has pages specifically on real examples of bad writing (especially by government employees)
  - It also has pages of how to guidelines and manuals. My favorites:
    - [Plain English at a glance](#)
    - [Tips on how to write good](#)
- Workable Web Solutions — a web site that has a reproduction of the original National Lampoon article by [Michael O'Donoghue on "How to Write Good"](#)

Write your reports for your peers: [other junior/senior chemistry students](#).

Yes, you may submit your lab reports electronically directly to your TA & Prof. However, even if you choose this option, you may also want to provide a written copy because what you submit *must* be in a standard format (Word or WordPerfect) that does not include idiosyncratic buried-fonts, characters, graphics, etc. that do not display correctly on someone else's computer. You will be graded on what displays on your TA's computer. Most appendix material will still have to be submitted the old-fashioned way unless it is done using a common application such as Excel.

A big help to preparing lab reports will be to have software that allows you to draw chemical structures and add them into your reports. You can obtain a [free copy of ChemSketch from ACD Labs](#). Go to the Dept. of Chemistry web page and choose [Chemistry Software](#) from the links at the bottom right of the page. Follow the directions.

Your first lab report will include a "do over" option. We realize that the first report you write may be a work in progress, and that

you will improve as you write more reports. Therefore, your first report can be revised and handed back in later for a regrade. **The caveat is that your revised first-lab report must be turned into your TA *before* that lab is presented as an oral presentation.** When you turn in your revised lab report, please include your original lab report too.

<b>Grading Rubric for Lab Reports</b>	
	<b>Points</b>
Abstract	3
Introduction	3
<b>Experimental</b>	
Description of instrumentation & procedures used	2
Definition of terms & equations used to perform calculations	5
<b>Results</b>	
Written explanation	5
Tables & Figures	10
<b>Discussion</b>	10
Literature cited	2
Appendix material	5
Overall composition and writing style	5
<b>Total:</b>	<b>50</b>
<i>Bonus:</i> Analyses that go beyond the required scope of the report	5

Last modified January 16 2012 06:09 PM

# The University of Vermont

## CHEM 201 Advanced Chemistry Laboratory

### Oral Presentation: What is required for Chem 201

Instructions for preparing your oral presentation are available for downloading as:

A [PowerPoint file](#)  
[PDF format](#)

#### Some ground rules for your oral presentation:

- There are 6 lab experiments that can be presented
- See the class schedule for the dates of the oral presentations
- You **cannot** present the first lab you perform – this lab must be a written lab
- Sign up is first-come, first-served, with the caveats:
  - All 6 labs will be presented (unless there is <6 students in the class)
    - If there are more than 6 students, then one or more labs may have 2 students signed up (but no more than two)
  - Send Prof Matthews and e-mail with the lab you want to present. If the lab is available and there are no other problems (see above), the lab is yours to present.

#### Grading Rubric for Oral Presentations:

	<b>Points</b>
Abstract	3
Introduction	5
<b>Experimental</b>	
Description of instrumentation & procedures used	3
Definition of terms & equations used to perform calculations	2
<b>Results</b>	
Clear presentation of key results	5
Tables & Figures	5
<b>Discussion</b>	10
Literature cited	2
Appendix material	5
Overall composition and speaking style	5
Quality of graphics	5
	<b>Total: 50</b>
<i>Bonus:</i> Analyses that go beyond the required scope of the report	5

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