Fall 2013
CH4212 - Instrumental Analysis Lab
Department of Chemistry
Michigan Technological University

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Teaching Assistants:

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Course Objectives:
(1) To gain a working knowledge of many of the instrumental analysis methods used in a modern chemistry lab.
(2) To provide the student with practical experience in the techniques of experimental, instrument-oriented, analytical chemistry.
(3) To deepen the student’s understanding of the underlying principles of the measurement by various laboratory instruments.
(4) To develop more sophisticated instrument skills in the student.
(5) To increase the student's ability to communicate effectively and professionally both in writing and in speech

Textbook (for CH4212):
Missed lab / tardiness policy:

YOU MUST TRY TO ATTEND ALL SCHEDULED LABORATORY MEETINGS. If you have to miss a lab due to illness or other emergency, please contact TA to validate the reason for being absent from the lab and get permission for the makeup. There will be one make-up laboratory session at the end of the term. If you miss a laboratory meeting, you must make it up during the same week or during the last week of labs. **If you miss two or more labs, you will fail the course.**

Tardiness: If you are more than 15 minutes late to lab, you will be marked late, and may be told to leave, depending on how late you are. If you are repeatedly late, you will fail the course. **All labs must be conducted and lab reports completed to pass the course (CH4212).**

Safety:

Corrosive, toxic, and flammable chemicals are used in this class. You must use good judgment when handling all chemicals, e.g. use the hoods when working with volatile substances or gloves when using toxic chemicals. Specific hazards regarding the chemical instrumentation are presented in each lab handout. **You are required to have chemical splash safety goggles or safety glasses.** I also recommend a lab apron or lab coat, since you will be handling strong acids and bases in this course. You will need a bound laboratory notebook for recording data. Please note locations of the eyewash and shower stations. Cleanliness is also an important aspect of a safe laboratory. Keep the work areas clean! Unsafe lab practices could result in a deduction in your final course grade. Goggles and lab coats are available at the chemistry stockroom. You will need the goggles and notebook for the first lab period.

Other Stuff:

You will need some sort of data storage medium (e.g. flash memory) for saving programs and spreadsheet files. Do not rely on the lab computers for storage. Files may be inadvertently deleted. Lastly, I appreciate your help in maintaining the lab. Please keep the printers loaded with paper, throw away (or recycle) used paper, fill distilled water (DW) bottles, etc.

Preparation:

The key to efficiency in the lab is preparation. You are expected to read the lab and contact the TA before the lab period for questions and/or clarifications. We highly recommend that you consult with your group members before the lab period to divide responsibilities so that your laboratory time is used most efficiently. It is also important that you are considerate of your lab and must arrive at lab on time and be ready to begin work immediately. Before coming to lab, you must have read over the webnotes (see below) for the day’s experiment and any applicable information in the text. Otherwise the TAs will be instructed not to let you begin experimental work. Being tardy, working on a previous week’s lab report during class, not reading the lab manual, printing the lab manual during lab, or having an unexcused absence will automatically result in a preparedness score of 0. Good preparation will pay dividends as you will finish your lab work much more quickly and you will likely achieve better scores on your lab reports.

Webnotes: The webnotes are the lab manual in this class: available in Canvas. The Webnotes provide you with specific information about the experiment that will be performed, as well as background on the methods and important safety information. You can print all of
this information out on your computer at home and produce a “hardcopy” version if you wish, but I think you will find this to be unnecessary. In reading the webnotes, you can extract the essential procedural details and safety information for your notebook, while familiarizing yourself with the theory behind the experiment.

**Spreadsheets:**

As part of the preparation for lab, you will be required to prepare a data analysis spreadsheet in Excel or some other data analysis/presentation program. You will find that this step streamlines and simplifies the data workup and makes report writing easier. There are several workstations available for your use in the computer lab; but if you have a laptop computer, it is useful to bring it to this class. (It should be possible to keep it away from the chemical hazards in the lab.) Graphical results will only be accepted in computer generated format (no graph paper) merged into the lab reports.

**Lab notebook:**

A hard bound scientific lab notebook is required for the lab. Notebooks are available at the bookstore. **All data must be recorded in the laboratory notebook, not on loose sheets of paper or on the Lab manual.** You are required to keep a laboratory notebook throughout the term and will hand it in for a grade near the end of the semester. You will need a hand-written synopsis of the procedure for each experiment to work from and spaces for the data and observations to be recorded. If your computer crashes (heaven forbid), you should be able to write most of the lab reports based on the information in your notebook.

The primary purpose of the laboratory notebook is to record data and experimental details, especially those that are not included in the lab manual or are deviations from the lab manual. It is good practice to write detailed procedures, but it is not required. The notebook will be graded based on the following guidelines:

1. Your name and contact information on the outside cover.
2. The notebook has the few pages reserved for a table of contents. This table of contents must be kept up to date.
3. Title and date of lab. Also date all additional work if completed on different days. You should take notes in the lab notebook when the lab overview is presented.
4. Fill in all of the information boxes on each page.
5. No pages should be removed for any reason.
6. Record any changes in the procedure given in the lab manual.
7. All data entries must be in indelible (i.e. non-erasable) ink.
8. Record all raw data in an organized fashion. Mistakes and errors should be crossed out with a single line. White-out is not allowed.
9. Try to keep the notebook reasonably neat – you should be able to understand each entry. It is often helpful to construct tables prior to lab and fill them out as you collect data.
10. Record all relevant observations
11. Include chemical calculations (e.g. for making up standards, etc).
12. Keep your notebook current. It is unethical (not to mention illegal in many circumstances) to go back and fill in your notebook with data you wrote on loose pieces of paper. Backdating (i.e. going back and writing the date you think the data was recorded) is also forbidden.

Reports and Grading:
This laboratory is an official writing intensive course. You are encouraged to work with your partners on analyzing your data. However all written work must be individual efforts. Each student is assigned to present reports for all experiments. All students are expected to write his/her report in his/her own words (i.e., all calculations, data analysis, and write-up must be done independently) with your own calculations and interpretations. Copied reports will be penalized severely. In addition to completing the labs and obtaining and evaluating results, you will be expected to describe your work in reports that are similar to scientific journal articles. The TA will give you more information (and hands-on help if you need it) about the expectations of each lab report, as well as feedback on reports that have been submitted, to help you improve. Each lab report is worth 100 points. The late penalty is 10% per school day (maximum 50% deduction).

Lab reports will not be accepted from anyone that missed an experiment. You may not simply copy your partners’ data and turn a report; you must arrange to do the experiment (preferably during another lab period).

Academic Dishonesty:
A student who, in connection with his or her studies, disrupts a class, plagiarizes, cheats, or otherwise violates reasonable standards of academic behavior may, at my discretion, have his or her enrollment canceled and/or be given a reduced or failing grade. For more information on specific acts that can constitute academic dishonesty, see your student handbook.

Disabilities:  
Any student who, because of disability, may require some special arrangements in order to meet course requirements should contact the instructor or TA as soon as possible to make necessary accommodations.

LABORATORY MODULES  
The laboratory course is divided into five modules, each of which emphasizes a different aspect of instrumental analysis. Each module consists of 1-3 experiments that relate to the theme of the module. You will work as part of a 2- or 3-person group.

Course Outline:

<table>
<thead>
<tr>
<th>Week</th>
<th>Starting date</th>
<th>Experiment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>September 2, 2013</td>
<td>No lab</td>
</tr>
<tr>
<td>2</td>
<td>September 9, 2013</td>
<td>Liquid Chromatography (LC)</td>
</tr>
<tr>
<td>Date</td>
<td>Event</td>
<td></td>
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<tr>
<td>--------------------</td>
<td>----------------------------------------------------</td>
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<tr>
<td>September 16, 2013</td>
<td>Gas Chromatography (GC)</td>
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<tr>
<td>September 23, 2013</td>
<td>Electrospray Ionization Mass Spectrometry (MS)</td>
<td></td>
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<tr>
<td>September 30, 2013</td>
<td>Matrix-assisted Laser Desorption/Ionization Mass Spectrometry (MS)</td>
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<tr>
<td>October 7, 2013</td>
<td>Gas Chromatography-Mass Spectrometry (GC/MS)</td>
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<tr>
<td>October 14, 2013</td>
<td>Liquid Chromatography-Mass Spectrometry (LC/MS)</td>
<td></td>
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<tr>
<td>October 21, 2013</td>
<td>Ultraviolet Spectrophotometry</td>
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<tr>
<td>October 28, 2013</td>
<td>Atomic Absorption Spectrophotometry</td>
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<td>November 4, 2013</td>
<td>Atomic Emission Spectroscopy</td>
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<td>November 11, 2013</td>
<td>Infrared Spectroscopy</td>
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<td>November 18, 2013</td>
<td>Fluorescence Spectrometry</td>
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<tr>
<td>December 2, 2013</td>
<td>NMR Spectroscopy</td>
<td></td>
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<tr>
<td>December 9, 2013</td>
<td>The make-up lab</td>
<td></td>
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</tbody>
</table>

The above schedule represents a first guess. Be ready to change and be flexible as the semester progresses.

**Summary of lab report requirements**

Laboratory reports must be prepared using a word processor, single sided only, with numbered pages, and stapled in the upper left corner for hard copy. Reports should be well organized and written as concisely as possible but using full sentences throughout. Adding lots of extraneous information won’t help (and might hurt) your grade. You can work together with your lab partner but each of you must create your own plots and write your own reports. Identical figures, tables or wording will lose 10 pts (of 100 pts total) for each report.

Reports must be printed with a high quality printer and have a title page containing the information shown on page ix. One member of your group must attach your copies of the raw data.

Make a cover sheet for every report. See the example on page ix.

**On every page of the report** include a header or footer with the page number and your name.

1. **Title Page.**
   Your lab report must have the title page cover containing your name and section number, the experiment number and name, date that the experiment was conducted, and date of report.

2. **Abstract.**
   The abstract should be a short paragraph describing the purpose, technique, and results of the experiment—think of the abstract as a concise summary.

3. **Introduction.**
   This short section should be 1 paragraph at most. It should briefly describe the technique(s) employed in the experiment (in your own words) and provide justification as to why it works. Cite any sources used.
4. Procedure.
If there were no changes to the procedure published in the lab handout, you can simply refer to that procedure in this section. If there were changes, then you must document those differences here in your own words and in narrative form. For example, "The procedure in the laboratory handout was employed with the following exceptions: ...". Between what you write and what is in the handout, someone should be able to repeat your experiment.

5. Data.
Present all your raw data (weights, volumes, instrument readings, etc.) using tabular and, if appropriate, graphical form. Graphs and tables should be clearly captioned. Axes should be clearly labeled. Units must be clearly stated and consistency of significant figures maintained.

6. Calculations.
Show all steps, including units, in your calculations. Show an example of each calculation performed. Pay attention to significant figures.

7. Results.
This section should contain a clear tabular and/or graphical report of the outcome of your calculations. Tables and graphs must each have a title and contain all pertinent data (e.g., concentrations of stock solutions, wavelengths, etc.). All values should have units and be presented to the correct number of significant figures. Straight line data should be subjected to least squares analysis (included in the spreadsheet software). The original output from the experiment must be included in the report of one member of the team.

Your data need to be well organized so that it is clear what you’ve done. Always organize this section following the order information is requested in the Lab directions. Don’t include any extraneous tables or numbers (e.g. data that are shown in a plot usually do not need to be tabulated). Take care to show the proper units and significant figures when reporting results!

Plots can be printed directly from the instrument software or spreadsheet. They may be integrated directly into the discussion or plotted as separate pages, following the section in which they are discussed (not all stapled at the back of the report). Take care to label plots properly. Use appropriate axes ranges that clearly show the variability in the data.

When discussing results refer to specific figures and tables (e.g. “the analyses were very reproducible as can be seen in Table 1”).

8. Discussion.
This section should explain your results and how they support (or not) the concepts of the experiment. Do your results agree with the expected literature results? Include observations noted during the experiment. Were there any problems with the experiment? How can the experiment be improved? How did these factors impact your experiment? Define any terms or concepts you use. Be sure to answer all questions asked in the lab handout. Explain sources of error in your experiment, but do not just make up problems that might have affected the results. If appropriate, compare your results to expected values and report the percent error (% Error = Absolute Value (((Observed - Expected)/Expected)*100)).

9. Conclusions:
In this section the overall results of the analysis should be restated and, whenever possible, compared to literature values. Pertinent comments and observations about the results should be made, and major sources of error (including sources in addition to personal error) should be discussed. Include in this section any discussion requested in the lab manual for each experiment. Each report should include a detailed discussion of both random and systematic errors that could have occurred with the instrumental method being used (at least three sources of each). All systematic errors must include a discussion of how the error would affect the final results.

10. References
Special procedures, literature values, and discussions of previous research results must be referenced in the text using superscript numbers. The references themselves belong in a separate section at the end of the report using the format specified in the ACS Style Guide (see http://jchemed.chem.wisc.edu/Journal/Authors/References.html for details).

11. Division of responsibilities.
Each student must independently fill out a “Division of responsibilities” form for each lab report. Please see form of Division of Responsibilities in following page.

Additional comments
1. Everyone must do his or her own work. You may discuss calculations and conclusions, but you must do your own calculations, answer the questions, and write your own lab report.
2. All lab reports must be typed. Graphs must be computer generated. For details on how to insert figures into Microsoft Word without creating a megabyte monster of a file. Equations should be entered using the Microsoft Equation (Insert > Object > Equation).
3. At this level, lab reports should show understanding of what you are doing and why.
4. Proof read your lab reports. Grammar and spelling should be perfect.
5. Lab reports are due at the start of lab one week after completion of each experiment. Hard copy and electronic copies must both be turned in to the TAs. Please either email your report to TA or bring a CD or memory stick containing your report to the TA on the due date. Make sure that your report is labeled with your name, experiment number, and date. All lab reports should be submitted electronically by either (a) emailing the Word document and any associated Excel spreadsheets to your TA.
6. Late lab reports will be penalized one letter grade for every day late. No lab reports will be accepted after the last day of classes.
7. Do not miss lab. If you must miss a lab, contact your TAs by email or phone prior to the lab to reschedule. You are not guaranteed that a lab can be rescheduled. All labs must be conducted and lab reports completed to pass the course (CH4212).

Lab report grades will be based on correctly following the lab directions, correct chemical calculations, clear and concise presentation and interpretation of results, and correct lab format. In cases where you analyze an unknown, some of your grade will be based on the precision and accuracy of the analysis and discussion of possible errors.
Division of Responsibilities

Each partner must turn in a separate copy of this form to the TA on the day the report is due. This form is to be filled out without collusion or discussion among partners.

My name:________________________________________

CH4212 Section:__________________________

Experiment number:_________________________

My contribution to this experiment (lab activities) is (percentage):_____

Name of my partner__________________________

If you had more than one partner, fill in your evaluation of each.

Name of my partner

My estimate of my partner’s contribution to this report (percentage): ______

Total 100 %

________________________________________  _________________________
Signature                        Date