Facility: VEICLE Lab (19-708) and 19-408
Class Hours: Lecture - 9 AM MWF; Laboratory – 1-4 PM T, R
Lecturer: D. J. Chesney, office 19-404C; phone 370-7405; email djchesne@mtu.edu
Office hours: By arrangement (email works well)
4222 Web Page: http://chemistry.mtu.edu/~djchesne/classes/ch4222/

The solutions manual for this text should be available online if you wish to purchase it.

Prerequisites: Chemistry 1120, 3510, 3511. A firm grounding in ionic equilibria is assumed and will be essential for success in this course. You should review the appropriate material from CH 1120 and Harris, Chapter 6.

Resource materials: Solved exams and quizzes from a previous edition of CH 2212 will be available on the course web site. These are intended to indicate the level and type of question your lecturer is likely to use, but no guarantee is made that quizzes this term will be in any way identical.

The laboratory portion of the course consists of experiments described in the lab handouts.

Evaluation:

<table>
<thead>
<tr>
<th>Component</th>
<th>Points</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quizzes (best 12 of 13):</td>
<td>240 pts</td>
</tr>
<tr>
<td>Homework</td>
<td>280 pts</td>
</tr>
<tr>
<td>Final Exam:</td>
<td>200 pts</td>
</tr>
<tr>
<td>Spreadsheet homework (4)</td>
<td>80 pts</td>
</tr>
</tbody>
</table>

Quizzes. A 20-point quiz will be given in lab every Tuesday this session (except for the first week). The lowest quiz score will be dropped. A missed quiz will be graded as a zero and can be used as the score to drop. Any topic previously covered in the course is fair game for a quiz question. The chemistry relevant to laboratory work may appear on quizzes and the final exam.

All work must be shown on the quizzes. However, only the answers will be graded.

For many of the quizzes, the use of calculators will not be allowed. The final problem set up and a reasonable estimate of the answer will be the answers to be graded in these quizzes.

Homework. Each week’s assigned problems must be turned in the following Monday at the beginning of class (9 AM). No late submissions will be accepted. Only one problem from each homework set will be graded - this problem will be selected randomly and will be worth the entire 20 points. Failure to turn in a completed problem set will result in a proportional deduction from your score. I.e., you got all 20 points on the graded problem but you only turned in 6 completed problems from an 8-problem set. Your score then is $20 \times \frac{6}{8} = 15$.

Final Exam. The comprehensive final exam will be given at the time determined by the scheduling office during finals week.

Grading: Straight scale (A, 90%; B, 80%, etc.) The lecture and laboratory grades will be scaled such that they contribute proportionally to your final grade: 60% Lecture, 40% Laboratory.

Assignments for the term are given in the following course schedule. Note the Summary at the end of each chapter. It is strongly suggested that you test your knowledge by trying Exercises and Problems at the end of each chapter in addition to those assigned below; you should also review the Terms To Understand listed for each chapter. Note that solutions to selected Problems are given in the back of the text.
**Course Schedule:** It’s a plan, not a contract.

<table>
<thead>
<tr>
<th>Week of</th>
<th>Read</th>
<th>Do</th>
</tr>
</thead>
</table>
| Jan 14  | Chapter 0 - The Analytical Process  
Chapter 1 – Measurements  
Chemical concentrations  
  Molarity, molality, weight percent, ppm  
Preparing solutions  
Calculations, lab exercises  
Chapter 2 - Tools of the Trade  
Lab notebooks  
Balances  
  Analytical vs top-loaders  
Volumetric glassware  
Pipets and micropipets  
Filtration  
Drying | | |
| Safety | Handling/storing chemicals responsibly in the lab  
MSDS sheets/Right-to-know | |
| Balance Exercise | Proper use of the analytical balance; weighing by difference  
Proper use of the top-loading balance; use of weigh boats | |
| Jan 21 | MLK Day Monday | |
| | Chapter 3 - Experimental Error | p50: Exercises 3A-3C  
p50: Problems 3, 5-7,15,16 | |
| | Use/Calibration of Volumetric Glassware  
Calibration of Micropipets  
Use of Excel spreadsheets in the analytical laboratory | |
| Jan 28 | Chapter 4 - Statistics  4-1 – 4-6 (omit sect 4-5)  
Attn: Box 4-1, p.63 | p73: Exercises 4A  
p74: Problems 3,12,17,18, 20-22 | |
| Solution preparation | Use of glassware; Transfer techniques;  
When to use volumetric glassware and when not to  
When to use the analytical balance and when not to | |
| Statistics worksheet | Using canned data, students will perform the full range of statistical tests | |
| Feb 4 | Chapter 6 - Review of Equilibrium Calculations | |
| Solution preparation | Students will prepare solutions of copper sulfate by weighing and dilution to a specified range, starting with both the hydrated and the non-hydrated salt; Quantitative comparison to a calibration curve prepared by lab instructors. | |
| Feb 7-8 | Winter Carnival recess | |
Week of | Read | Do
--- | --- | ---
Feb 11 | Chapter 9 - Acid-Base Equilibria | p177: Exercises 9B,D,H,J
 | | P178: Problems 6-8,11,13, 20,23,24,32,40
**Equilibria worksheet I**

*Use and Care of pH probes/pH measurement*
Potentiometry, Activity and pH errors

Feb 18 | Chapter 10 - Polyprotic Acid-Base Equilibria | p195: Exercises 10A,B,C
 | | p196: Problems 4,5
**Equilibria worksheet II**

**Buffer Preparation I**
Use of the Henderson-Hasselbalch Equation to predict buffer composition;
Preparation of same

Feb 25 | Buffers of biological importance | p76: Problems 23, 29, 30
Chapter 4 - Statistics  4-7 – 4-8 | p92: Exercises 5A-C
Chapter 5 - Calibration Methods | p93: Problems 15,18, 19, 23, 28, 29
**Buffer Preparation II – Theory vs Reality**
Students will prepare biochemically significant buffers from recipes;
Measure resulting buffer pH to properly calibrated pH meter

**Preparation of constant ionic strength buffers**

Mar 3 | Chapter 18 - Spectrophotometry - Fundamentals | p398: Exercises 18A-D
 | | p399: Problems 6,10,12,16,19

**Tutorial on Ocean Optics Spectrophotometers**

**Calibration Curves/Standard Addition**
Students will be assigned to produce a linear calibration curve in a specific absorbance range by dilution of a stock solution; The standard additions method of quantitation will be illustrated with these same solutions.

**Production of the Perfect Isosbestic Point**
An exercise that combines preparation of buffers and dilution technique with spectrophotometric verification of results.

Mar 8 – Mar 17 | Spring Break
Mar 17 | Chapter 19, 20 – Spectrophotometry – Hardware and Applications | p417: Exercises 19A-C
 | | p418: Problems 1,2,10,11
 | | p450: Exercise 20-A
 | | p450: Problems 3-6,8,10,16,17,27,28
Analyzing mixed components
Scatchard plots for equilibrium constants
Components of spectrophotometers, particularly those that are user-selectable
Spectrophotometric Determination of Iron in Dietary Supplements
Students will extract iron from a tablet with dilute acid, generate a colored complex by reaction with α-phenanthroline and quantitatively determine the iron present by comparing the absorbance to a calibration curve.

Spectrophotometric Analysis of a Mixture: Caffeine and Benzoic Acid in a Soft Drink
Students will learn how to select measurement wavelengths for analysis of individual components in a mixture; Students will also solve two equations for two unknowns using either matrix or algebraic methods.

Mar 24
Chapter 23 – Analytical Separations
p524: Exercises 23A-F
p525: Problems 1,2,4,9,11,19,21, 24,27,37,40

Enzyme Kinetics: Michaelis-Menten plots
Fluorescence titration
Students will use a fluorescence spectrophotometer for the quantitative determination of ligand-protein binding constants

Mar 31
Chapter 24 – Gas Chromatography
p551: Exercises 24A-E
p552: Problems 1-6,8,9,11,13,18

Use and care of GC; Methods development
Separation of fat-soluble vitamins by GC;
Analysis of a commercial multi-vitamin product

Apr 7
Chapter 25 – High Performance Liquid Chromatography
p584: Exercises 25A-D
p585: Problems 6,7,15,17,18,23,29, 30,31,33,34

Use and care of HPLC; Methods development
Separation of water-soluble B vitamins by HPLC;
Analysis of a commercial multi-vitamin product

Apr 14
Chapter 26 – Chromatographic Methods
p623: Exercises 26A,B
p624: Problems 5,6,11,16-19,21

Separation of enantiomers by chiral HPLC

Apr 21
Chapter 22 – Mass Spectrometry
Mass Spectrometry demonstration
Laboratory Component

Introduction:

Laboratory experiments and exercises are indicated in **bold** in the schedule above. Graded lab experiments are **underlined**. Instructions appropriate to the lab experiment or exercise will be provided on the course web page. **These instructions will not be available on the VEICLE Lab computers.** No printed copy of these instructions may be brought into the lab. You must prepare your own experiment instructions in your lab notebook before you come to lab. A copy of these instructions must be turned in at the beginning of each experiment.

All preliminary calculations contained in the lab handouts must be done in the lab notebook on a page separate from the experimental procedure. A copy of these preliminary calculations must be turned in to the laboratory instructor prior to initiating the experiment.

Lab Notebooks:

Part of the message in this lab is to teach you to keep a proper analytical notebook. **A bound notebook with carbonless copies is required.** Complete guidelines have been published (“CH 2212 Notebook Format”, see the Laboratory section of the web page). Keep your notebook complete, readable, and up-to-date!

Lab Reports:

Lab results are due at the beginning of the second lab period following the normal scheduled end date of the experiment. The lab report will consist solely of the copies of your lab notebook, unless specifically directed otherwise. The summary page in your lab notebook corresponding to each experiment will be the primary reporting vehicle. Additional charts, graphs, etc., may be asked for in a given lab exercise, and these should be appended to the notebook pages. Obviously, if I can’t follow your notebook pages, I can’t give you a grade.

Graded Lab Experiments and Exercises:

<table>
<thead>
<tr>
<th>Experiment Description</th>
<th>Points</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calibration of Micropipets</td>
<td>100 pts</td>
</tr>
<tr>
<td>Use of Excel spreadsheets in the analytical laboratory</td>
<td>100 pts</td>
</tr>
<tr>
<td>Statistics worksheet</td>
<td>100 pts</td>
</tr>
<tr>
<td>‡Solution preparation</td>
<td>100 pts</td>
</tr>
<tr>
<td>Equilibria worksheet I</td>
<td>100 pts</td>
</tr>
<tr>
<td>Equilibria worksheet II</td>
<td>100 pts</td>
</tr>
<tr>
<td>Buffer preparation I</td>
<td>100 pts</td>
</tr>
<tr>
<td>Buffer preparation II</td>
<td>100 pts</td>
</tr>
<tr>
<td>Production of the perfect isosbestic point</td>
<td>100 pts</td>
</tr>
<tr>
<td>‡Spectrophotometric Determination of Iron in Dietary Supplements</td>
<td>100 pts</td>
</tr>
<tr>
<td>‡Spectrophotometric Analysis of a Mixture</td>
<td>100 pts</td>
</tr>
<tr>
<td>‡Enzyme kinetics</td>
<td>100 pts</td>
</tr>
<tr>
<td>‡Fluorescence titration</td>
<td>100 pts</td>
</tr>
<tr>
<td>‡Separation of fat-soluble vitamins by GC; Analysis of a commercial multi-vitamin product</td>
<td>100 pts</td>
</tr>
<tr>
<td>‡Separation of water-soluble B vitamins by HPLC; Analysis of a commercial multi-vitamin product</td>
<td>100 pts</td>
</tr>
<tr>
<td>Separation of enantiomers by HPLC</td>
<td>100 pts</td>
</tr>
</tbody>
</table>

‡Quantitative experiment

Total: 1600 points, which will be scaled to be numerically equal to forty (40) percent of your CH 4222 course grade.
CH 4222 Laboratory grading procedure:

Because many of these experiments are new, we do not have statistical data from previous classes. Also, many of these experiments are “less than quantitative” in nature, requiring a different approach to grading than that used in our traditional Quantitative Analysis course. Hence, for those quantitative determinations that we can control, I will base the grading on the standard deviation of the results reported from this class (ignoring the obvious outliers).

Worksheets, of course, will be graded on number correct. Spreadsheet assignments will be based on my famous totally subjective scale.

No formal lab reports will be involved. Your entire score for a given exercise or experiment will be based on the lab notebook copies you turn in, along with any pertinent supplementary material that I specify. Your results are due by the beginning of the second lab period following the end of your scheduled time for completion of the experiment. Exceptions will be made for health and other valid reasons, but must be reported to your TA promptly. Failure to turn your results in on time will result in a grade of zero for the experiment in question.

If you have made a calculation error in your submission, you may re-calculate and submit with a thorough explanation. Only one re-calculation will be allowed per quarter.

Experimental results breakdown:

<table>
<thead>
<tr>
<th>Category</th>
<th>Points</th>
</tr>
</thead>
<tbody>
<tr>
<td>Readability/legibility/logic of notebook</td>
<td>25 pts</td>
</tr>
<tr>
<td>Complete summary page(s) for experiment</td>
<td>25 pts</td>
</tr>
<tr>
<td>Data (Qualitative, subjective)</td>
<td>50 pts</td>
</tr>
<tr>
<td>Data (†Quantitative)</td>
<td></td>
</tr>
<tr>
<td>$\mu = \text{known value or label value}$</td>
<td></td>
</tr>
<tr>
<td>$s = \text{class standard deviation}$</td>
<td></td>
</tr>
<tr>
<td>$\mu \pm 0.3 \ s$</td>
<td>50</td>
</tr>
<tr>
<td>$\mu \pm 0.6 \ s$</td>
<td>40</td>
</tr>
<tr>
<td>$\mu \pm 0.9 \ s$</td>
<td>30</td>
</tr>
<tr>
<td>$\mu \pm 1.2 \ s$</td>
<td>20</td>
</tr>
<tr>
<td>$\mu \pm 1.5 \ s$</td>
<td>10</td>
</tr>
</tbody>
</table>
Class Schedule And Absence Policy

**Quizzes:** The quizzes in this class are scheduled during lab hours in an attempt to minimize scheduling conflicts. An *unexcused* absence is an automatic zero for any quiz that is missed. An *excused* absence may be granted by the Office of Student Affairs *only*. If you know that you will have an official university excused absence on a day that a quiz is scheduled (university athletic event or religious holiday), you are required to make arrangements as early as possible *in advance of the quiz date*. Other examples of excused absences granted in the past are serious illness or a death in the family. Excused absences are not given to travel home or to attend a social event. Plan to take your quizzes at the scheduled time.

**Labs:** You cannot imagine the degree of difficulty involved in making up a lab. The criteria for absences are the same as in the preceding paragraph regarding quizzes.

**Academic Integrity**

Standards of academic conduct are set forth in the University's Academic Integrity Policy, which can be found at [http://www.studentaffairs.mtu.edu/dean/judicial/policies/academic_integrity.html](http://www.studentaffairs.mtu.edu/dean/judicial/policies/academic_integrity.html). Specific violations in this course would be the intentional use of any unauthorized study aids, equipment, or another’s work during an examination (cheating) or allowing/helping another individual to cheat (facilitating academic dishonesty). Possible sanctions include an academic integrity warning, an “F*” grade indicating failure due to academic dishonesty, suspension or expulsion.

Having said this, I acknowledge that one of the best ways to learn material is to try and explain it to someone else. The only opportunity you get to do that in this class is in doing homework assignments. So let’s be clear – I don’t have any problem with group work on homework assignments, with the understanding that you are actually teaching each other and learning in the process. **Each student still has to turn in his or her individual work, in his or her own writing.** And, of course, it is not acceptable to electronically copy a spreadsheet or any other digital document and stick your name on it. That will get you a trip to the Dean of Student’s office.

MTU complies with all federal and state laws and regulations regarding discrimination, including the Americans with Disabilities Act of 1990 (ADA). If you have a disability and need a reasonable accommodation for equal access to education or services at MTU, please call Dr. Gloria Melton, Dean of Students at 487-2212.