Introduction to CHEM-4181

CU- Boulder

CHEM-4181
Instrumental Analysis Laboratory

Prof. Jose-Luis Jimenez
Spring 2007

Presentation is posted on course web page – mostly same info as lab manual

The People Involved

• Instructor: Jose-Luis Jimenez
  – Build and use instrumentation (MS) for a living
  – First time teaching this class
  – Office hours: Mon & Fri 12-1

• TAs:
  – Jesse Marcum (full-time)
  – Paul Nelson (half-time)
  – Patrick Veres (half-time)

• 23 students
  – Brief introductions
  – 3 sections
    • 7/9/9 students
    • Tue, Wed, Thu 12-6 pm
Objectives of the Course

- Review of:
  - Statistical data analysis
  - Scientific writing
  - (These were weak areas based on prior offerings, and you need to do them well for lab reports)
- Fundamental understanding of modern chemical analysis
- Hands-on experience with common instrumental techniques
  - Most basic, a few research-grade instruments. But mostly same procedures as more expensive ones
- Develop critical thinking and evaluation skills within a scientific framework

Materials for the Course

- Lab Manual in UMC bookstore
  - Read, lots of useful info beside experiment details
- Principles of Instrumental Analysis, Skoog, Holler, and Niemann, 5th edition
  - 6th edition just came out, also ok
- Laboratory notebook
  - Your choice but must be research-grade
- Email list: chem-4181@lists.colorado.edu
- Web page:
  http://cires.colorado.edu/jimenez/CHEM-4181/
Lecture Schedule

<table>
<thead>
<tr>
<th>Lecture Date</th>
<th>Topic</th>
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<tbody>
<tr>
<td>January 17 (W)</td>
<td>Introduction</td>
</tr>
<tr>
<td>January 19 (F)</td>
<td>Data Evaluation and Statistics</td>
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<tr>
<td>January 22 (M)</td>
<td>Data Evaluation and Statistics</td>
</tr>
<tr>
<td>January 24 (W)</td>
<td>Data Evaluation and Statistics</td>
</tr>
<tr>
<td>January 26 (F)</td>
<td>Scientific Writing</td>
</tr>
<tr>
<td>January 29 (M)</td>
<td>Scientific Writing</td>
</tr>
<tr>
<td>January 31 (W)</td>
<td>Scientific Writing</td>
</tr>
<tr>
<td>February 2 (F)</td>
<td>No Class</td>
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<tr>
<td>February 5 (M)</td>
<td>Highlights for Lab Experiments</td>
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<tr>
<td>February 7-23</td>
<td>No Class</td>
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<tr>
<td>February 26 (M)</td>
<td>Introduction to Student Choice Experiment (Sci. Lit.)</td>
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<tr>
<td>February 28-29</td>
<td>No Class</td>
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<tr>
<td>March 5 (M)</td>
<td>Highlights for Lab Experiments</td>
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<tr>
<td>March 12 (M)</td>
<td>Student Choice Experiment Discussion</td>
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<tr>
<td>March 14-16</td>
<td>No Class</td>
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<tr>
<td>March 16 (F)</td>
<td>Student Choice Exp. Approval (in person or e-mail)</td>
</tr>
<tr>
<td>March 21 (W)</td>
<td>Student Choice Proposal Due</td>
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<tr>
<td>March 23 (F)</td>
<td>No Class</td>
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<tr>
<td>March 26 – 30</td>
<td>Spring Break, No Class</td>
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<tr>
<td>April 2 – April 13</td>
<td>Crafting a Scientific Presentation</td>
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<tr>
<td>April 18 &amp; 20</td>
<td>No Class</td>
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<tr>
<td>April 23 (M)</td>
<td>Student Choice Presentations</td>
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<tr>
<td>April 25 (W)</td>
<td>Student Choice Presentations</td>
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<tr>
<td>April 27 (F)</td>
<td>Student Choice Presentations</td>
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<tr>
<td>April 30 (M)</td>
<td>Student Choice Presentations</td>
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<tr>
<td>May 2 (W)</td>
<td>Student Choice Presentations</td>
</tr>
<tr>
<td>May 4 (F)</td>
<td>Student Choice Presentations</td>
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</tbody>
</table>

Lab Schedule

<table>
<thead>
<tr>
<th>Week Beginning</th>
<th>Experiment/Topic</th>
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<tbody>
<tr>
<td>January 15</td>
<td>No Lab</td>
</tr>
<tr>
<td>January 22</td>
<td>Introduction, Check In</td>
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<tr>
<td></td>
<td>Group Assignments</td>
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<td></td>
<td>Rotation Sign-Up (groups &amp; individuals)</td>
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<td></td>
<td>Glassware Cleaning</td>
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<td></td>
<td>Introduction to and Preparation for COD Exp.</td>
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<tr>
<td>January 29</td>
<td>(E1) Chemical Oxygen Demand</td>
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<tr>
<td>Feb. 5 – Feb. 26</td>
<td>(E2) Atomic Absorbance (Pb in soil)</td>
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<td>(E3) Molecular Absorbance (Phosphorous in water)</td>
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<td></td>
<td>(E4) Fluorescence (Oil in water)</td>
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<td></td>
<td>(E5) FTIR (CO in car exhaust)</td>
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<tr>
<td>Mar. 5 – Mar 19</td>
<td>(E6) GC (PAH’s in diesel exhaust)</td>
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<td></td>
<td>(E7) HPLC (PAH’s in cigarette smoke)</td>
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<td></td>
<td>(E8) Electrochemistry (metal ions in water)</td>
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<tr>
<td>March 26</td>
<td>Spring Break – No Lab</td>
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<tr>
<td>April 2</td>
<td>Student Choice Experiment</td>
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<tr>
<td>April 9</td>
<td>Student Choice Experiment</td>
</tr>
<tr>
<td>April 16</td>
<td>Finish Student Choice and Check Out</td>
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<tr>
<td>April 23</td>
<td>No Lab – Student Choice Presentations in Class</td>
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<tr>
<td>April 30</td>
<td>No Lab – Student Choice Presentations in Class</td>
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Lab sections

- Tue (7)
  - David Bishop, Chandra Avishek, Sarah McKenna, Estefania Mondragon, Benjaming Reed, Joseph Ryerson
- Wed (9)
  - Dane Clausen, Robert Ely, Kyle Galway, Maja Janas, Guillermo Rojas, Sally Sogue, Jung Song, Nicole Suiter, Asa Ware
- Thu (9)
  - Robert Busch, Rocky Draughon, Anne Getts, William Hartwig, James Kim, Michael Monsalve, Elizabeth Saenim, Elissa Uhl, Jennifer Fox
- Need a volunteer to move from Wed or Thu to Tue

Lab Experiments

- Read experiment description before showing up for lab
- Will work in groups
  - Arrive on time, otherwise team has to wait
  - Penalty if later than 12:15
- Reports are individual
  - Steep penalties for being late on reports (see LM)
A Good Lab Experiment

• Clearly Identify the question
• Qualitative vs. quantitative
• Determine most appropriate technique(s)
• Do you need to separate analyte from matrix?
• Acquire a representative sample

• See p. 7-8

Labeling and Waste Disposal

• Every solution that you prepare and which is not consumed that day should have a label
  – Photocopy from notebook, print from web
  – Paste with clear packing tape

• In order to receive a grade, you HAVE TO dispose of all waste according to EPA regulations
Grading

- Homework: 2 x 30 pts
- Lab reports: 8 x 60 pts
  - Submit to TA within 15 min of starting next lab
  - Steep penalties for being late (see LM)
- Weekly lab evaluations by TA
  - Performance = 8 x 10 pts (0 if late to arrive)
  - Notebook = 8 x 5 pts
- Student choice total = 210!
- Clickers: 30
- Bonus points for helping improve course

Your Lab Notebook

- Essential to keep a record of experiments for a scientist
  - Detailed enough for someone else to reproduce experiment
  - Especially important when intellectual property is an issue, or when authenticity is questioned
    - Dated, no blank pages
- To help you develop good notebook skills, TAs will check at the end of every lab
- See p.8 and p.59 for details and example
Clickers

• Will use for the data analysis & writing lectures
• No need to buy them
• Have 20 from Chemistry Dept.
  – Need to return them, otherwise charge $30 to your student account
• Does someone have an iClicker? (need 3)
• Grading for participation only
  – Will remove your worst clicker day
  – If you forget it more than once, you loose points

Your iClicker

Power Indicator (lighted when on)
Low Battery Indicator (<10 h left)
replace 3 AAA batteries before next class
Vote Status
  Green on for 0.5 sec to indicates vote received
  Flashing Red – not received
On-Off Button (keep on during class)
Have you used clickers in a prior class?

• A: Yes, iClickers
• B: Yes, but not iClickers
• C: No
• D: I can’t remember

You are NOT locked in to your first choice. Final answer entered will be the one accepted

Improving the Course

• Bonus for helping to improve the course
  – Finding mistakes in lab notebook (typos, errors)
  – Proposing improvements & tweaks to main experiments
  – Finding a useful web resource
  – Other useful suggestions (including alternative experiments)
  – If new & adopted, bonus of 1-5 pts per item (depending on how useful)
  – Email / talk to me about suggestions
Student Choice Experiments I

- Normally groups of 2 people (or individual)
- Should be a real problem
  - Environmental, food analysis
  - Has to be done with instruments in class, can’t use instruments available to you elsewhere
- Effort in creativity (e.g. caffeine in chocolate done every year)
- Procedures with extensive chemical workup are not appropriate (e.g. 1 page of workup in journal article => too much time)
- Must be planned and approved by March 16
  - Will discuss in class on March 12
  - But you can start thinking now (see example p. 66 of LM)

Student Choice Experiments II

- Timeline
  - 3 weeks to perform experiment
  - Lab open 9-5, but TAs there only during regular lab hours
  - Instrument schedule if several groups need to use same instrument
  - No lab after week of 4/23 (class presentations)
    - Some people earlier than others (lottery)
- Chemicals and Waste
  - Bill Eberle has Chemical inventory (x2-8973, Eberle@colorado.edu, Ekeley M240)
  - Can buy additional chemicals if needed (~$50 / group)
  - In proposal you should report the amount of waste that you will to generate
    - Managing waste is part of the grade
Questions?