LEAD LAB:
Community Based Exploratory Learning in the Context of Teaching Instrumentation With a Single Analyte

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8/01

Lead Lab: Teaching Instrumentation with a Single Analyte

"[The suspected material] is to be acted on with dilute nitric acid, and the resulting liquid filtered. If the addition of dilute sulphuric acid to the filtered liquid produce a white, if chromate of potash a yellow, and , lastly, if sulphuretted hydrogen a black precipitate, the presence of lead is fully determined."

Mitchell, 1848 A.D.: An Early Analytical Text

Take two to three pounds of the juice of the Lunaria, add a solution of lead, alum, salpetre, sal ammoniac, silver-litharge, quick-silver sublimate, vinegar and ginger, then distil and calcinate. This powder is then thickened with a silver solution into paste. One part in a hundred of this paste, heated on a copper plate, could theoretically turn tin, lead, or quicksilver into silver at a ratio of one to a hundred parts.

Raymond Lully, 1235 A.D.

Contents

*** not yet student proofed**

Key Concept Sequences

The following experiments have been tried and tested over a 6 year lab sequence for undergraduate chemistry majors. They are arranged in a historical and conceptual sequence allowing students to acquire a background understanding of chemistry.

Concept Sequence 1:

Sampling statistics control the **overall** analytical procedure beginning with in field sampling both in randomizing individual samples and in collecting a sufficient data base of information to derive meaningful information. Sampling statistics also control the **limit of detection**, a figure of merit used to select an instrument for use in analysis. By increasing the number of instrumental measurements using computer based collection of information, the standard deviation of the measurement goes down, and, therefore, so does the limit of detection.

Students are asked to make a running set of comparisons of the instrumental methods throughout the semester, so they need to be able to determine the **limit of detection** on each instrument and understand what factors come into play. **Experiments 1 and 2** set the student up to properly understand statistics and to use computer spread sheets, as well as some simple digital and electronic filtering to reduce noise.

Concept Sequence 2:

The final analysis is dependent upon the weakest link. Often that weak link is the analyst's understanding of the chemistry that precedes the instrumentation. One recurring theme in lead analysis is the need to control the pH in order to avoid lead hydroxide formation. Students are asked to build on the spread sheet skills in producing an alpha plot for lead hydroxides and to compare that data to measured free lead vs pH (**ISE, experiment 8).** Chemistry continues to be introduced in the form of chelation and phase separation in the UV-Vis Dithizone experiment. If the pH is not properly controlled the experiment can not work. The chemistry in this experiment also depends upon selectivity in chelation and on phase separation, concepts that reappear in the IR, NMR, and Chromatographic experiments.

Concept Sequence 3:

Lead exists in a variety of isotopes. The NMR and MS labs exploit the fingerprint pattern of lead.

Concept Sequence 4:

Ethics is implicitly and explicitly built into this lab. **Explicit** ethical issues to be explored include "professional" ethical issues relating to professional relationships within the work groups, honesty, integrity (Lab 1: a good lab book; Lab 23: digestion and designing internal quality control). We also explore how statistics (Lab 1) informs public policy in a different manner than it does analytical measurements. We directly explore (last lab) how a scientific disagreement should be carried out. **Implicit** ethic issues concern ownership of data between unequal collaborators (community groups and scientists), the extent to which scientists have a moral obligation to impart scientific knowledge to the general public, and the obligation that scientists have to not participate in scare science and/or to "use" community groups as mere learning tools.

Part A:
Class Syllabus and Information

Instrumental Methods of Analysis
Chemistry 310/311
Fall 2001
Loyola University Chicago.

Goal: To be able to chose an instrument for lead analysis using limits of detection, and, reliability, and ease of use. Problem Based Learning to monitor house dust with collaborating 6th grade children or to produce a publication quality research project on lead around a municipal waste incinerator.

Things students will be graded on:

Exams (4), all must be taken, however lowest grade may be averaged with highest to get a value for the fourth exam . 400 points

Informed Class participation (20%) 100 points

A:	90%
B	80- 89.9%
C.	70-79.9%
D.	60-69.9%

Text Teaching Instrumentation with a Single Analyte (Fitch)
Optional: Skoog, Leary, Nieman 6th Ed.

EXAM FORMAT

3 essays, 4 problem solving
Problems derived from homework and from lab
Essay may include reading a contemporary article

List of Equations on the following pages may be xeroxed, highlighted, **but not annotated**, and brought to exams.

CHEAT SHEETS

This may be xeroxed & brought to exams. You may use a highlighter on this sheet only.

Constants

h = 6.62608x10^{-34} J-s

k = 1.38x10^{-23} J/K

c = 3x10^{8} m/s

R = 8.31451 J/mol-K

F = 9.6485x10^{4} C/mol

N = 6.022x10^{23} particles/mole

The Golden Key: The Normal Equation

$$f(x) = \frac{1}{s\sqrt{2p}} e^{-\frac{1}{2}\left(\frac{x-m}{s}\right)^2}$$

Electronics and Noise and Statistics

V=IR

V = q/C

dq/dt = I

V_o = -â(å$_-$ + å$_+$)

$$X_c = \frac{1}{2pfC}$$

$$Z = \sqrt{R^2 + X_c^2}$$

$$\Delta f = \frac{1}{t_r}$$

$$R = \frac{\left(\bar{x}_a - \bar{x}_b\right)}{6s}$$

$$\frac{S}{N} = \frac{\bar{x}_a - \bar{x}_b}{s}$$

$$LOD = x_{blank} \pm 3s_{blank}$$

$$LOQ = x_{blank} \pm 9s_{blank}$$

$$6s \approx \left|Peak_{\max}\right| - \left|Peak_{\min}\right|$$

Optics

$$nl = c$$

$$E = hn$$

$$h_i = \frac{c}{n_i}$$

$$f = \frac{n_m 2n}{c}$$

$$l = \frac{2th}{n}$$

f =F/d

$$W = \frac{\Delta l}{2D^{-1}}$$

$$l_{\max} = \frac{2898}{T}$$

$$nl = d(\sin i + \sin r)$$

$$J_l = \left(\frac{2phc^2}{l^5}\right)\left(\frac{d_l}{e^{\frac{hc}{kTl}} - 1}\right)$$

$$Pa\left[\frac{1}{f/\#}\right]^2$$

$$R = nN$$

$$\frac{\sin q_1}{\sin q_2} = \frac{h_2}{h_1}$$

$$\frac{I_R}{I_o} = \left(\frac{h_2 - h_1}{h_2 + h_1}\right)^2$$

$$I_s = \left[\frac{I_o}{l^4}\right]\left[\frac{8p^4 a^2}{r^2}\right]\left[1+\cos^2 q\right]$$

Spectroscopy (100-800 nm)

$$A = -\log T = -\log\left(\frac{P}{P_o}\right) = \log P_o - \log P = ebC$$

$$A = \log\left[\frac{P_{o,l_1} + P_{o,l_2}}{P_{o,l_1}\left(10^{-e_1 bC}\right) + P_{o,l_2}\left(10^{-e_2 bC}\right)}\right]$$

$$\frac{s_c}{C} = \frac{0.434 s_T}{T\log T}$$

$$\left|\Delta l_{1/2}\right| = \frac{l^2 \Delta n}{c}$$

$$\Delta l = 2\left(\frac{l}{c}\right)\left(\frac{2kT}{pm}\right)^{1/2} = 4.848x10^{-7}\, l\sqrt{\frac{T}{m}}$$

$$\frac{N_j}{N_o} = \left(\frac{P_j}{P_o}\right)e^{\frac{-\Delta E}{kT}}$$

Spectroscopy > 1µ

$$m = \frac{m_1 m_2}{m_1 + m_2}$$

$$n = \frac{1}{l} = \frac{1}{2pc}\sqrt{\frac{k}{m}} = 5.3x10^{-12}\sqrt{\frac{k}{m}}$$

#bands possible: 3N-5 or 3N-6

$$\Delta N = 2b\left(n_1 - n_2\right)$$

$$b = \frac{\Delta N}{2\left(n_1 - n_2\right)}$$

Electrochemistry

$$\Delta G = -nFE = -RT\ln K$$

$$E = E^o - \frac{0.0591}{n}\log\frac{[\mathrm{Re}\,d]}{[Ox]}$$

$$E_{ISE} = const' + 0.0592\log\left(a_{analyte}\right)$$

$$E_{measured} = E_{cell} = E_{rhs} - E_{lhs} = E_{cat} - E_{an}$$

$$\frac{hc}{l} \propto \Delta E^o$$

$$i = nFAD\frac{\P C}{\P x} = nFAD\frac{\Delta C}{d}$$

where $d \approx \sqrt{2Dt}$

$$I_{limiting,RDE} = 0.620\, nFAD^{2/3} n^{-1/6} w^{1/2} C$$

$$I_{polarography} = 0.607\, nm^{2/3} D^{1/2} C_{bulk} t^{1/6}$$

$$i_c = AC_d\left[\frac{\P dE}{\P t}\right] = AC_d n$$

$$q = nFN$$

The Diffusion Equation: Does it Look Familiar?

$$C_{x,t} = \frac{C_o}{\sqrt{4pDt}} e^{-\left(\frac{x^2}{4Dt}\right)}$$

$$= \frac{C_o}{\sqrt{2Dt}\sqrt{2p}} e^{-\frac{1}{2}\left(\frac{x}{\sqrt{2Dt}}\right)^2}$$

Chromatography

$$k' = \frac{t_r - t_m}{t_m}$$

$$k' = \frac{KV_s}{V_M}$$

$$\mathbf{a} = \frac{t_{r,B} - t_m}{t_{r,A} - t_m} \qquad \mathbf{a} = \frac{K_B}{K_A}$$

$$N = \frac{16t_r^2}{W^2} \qquad H = \frac{L}{N}$$

$$H = \frac{W^2 L}{16t_r^2}$$

$$H = \frac{B}{u} + C_s u + C_m u$$

$$B = 2yD_m$$

$$C_s = \frac{qk' d_f^2}{(1 + k')^2 D_s}$$

$$C_m = \frac{f\left(d_p^2, d_c^2, u\right)}{D_m}$$

$$R_s = \left[\sqrt{\frac{N}{4}}\right]\left[\frac{\mathbf{a} - 1}{\mathbf{a}}\right]\left[\frac{k'_b}{k'_b + 1}\right]$$

$$t_{r,b} = \left[\frac{H}{u}\right] 16R_s^2 \left[\frac{\mathbf{a}}{\mathbf{a} - 1}\right]^2 \left[\frac{(1 + k'_b)^3}{k'^2_b}\right]$$

<table>
<tr><th colspan="6">Schedule: Alanah Fitch, Fall, 2001</th></tr>
<tr><th>Day</th><th>M</th><th>T</th><th>W</th><th>Th</th><th>F</th></tr>
<tr><td>8:30</td><td rowspan="4">Chem 311
Lab</td><td rowspan="4">Chem 311
Lab</td><td rowspan="4">Chem 311
Lab</td><td colspan="2">Babysitting</td></tr>
<tr><td>9:30</td><td></td><td rowspan="5">At Env.
Studies/Sci.
Program
Damen 111
phone: 88992

Office Hours</td></tr>
<tr><td>10:30</td><td></td></tr>
<tr><td>11:30</td><td>Chem.
Seminar</td></tr>
<tr><td>12:30</td><td>Lunch</td><td>Lunch</td><td>Lunch</td><td>Lunch</td></tr>
<tr><td>1:30</td><td rowspan="4">At Env.
Studies/Sci.
Program
Damen 111
phone: 88992
Office Hours</td><td></td><td rowspan="4">Chemistry
Research</td><td></td></tr>
<tr><td>2:30</td><td rowspan="2">Lecture
Chem 310</td><td rowspan="2">Lecture
Chem 310</td><td>Chem 101</td></tr>
<tr><td>3:30</td><td rowspan="2"></td></tr>
<tr><td>4:30</td><td></td><td></td></tr>
</table>

Office	Department	Phone	email
402 Flanner Hall	Chemistry	508-3119	afitch@luc.edu
111 Damen Hall	Environmental Studies/Sci.	508-8992	
	Home	847-256-8243 (between 6-9 p.m.)	

Office Hours: M 1:30 to 5
F 9:30-2

Revised Schedules: Fall 2000 Instrumental Analysis/ September 21, 2000

						Stat	Chem	Isotope	Ethics
1	Aug 29	T	Lead and Society	Red Potatoes, White Potatoes, Experiment 1 **Divide into Semester long Teams I and II** Alanah					
	Aug 31	Th	Statistics and Ethics						
2	Sept 5	T	Signals and Noise	Electronics: Analog and Digital Filtering Experiment 2 Alanah and Carla					
	Sept 7	Th	Signals & Noise, LOD						
3	Sept 12	T	Relevant **Chemistry**	Lead Hydroxide Equilibrium Chemistry and Ion Selective Electrodes Experiment 8 Jorge					
	Sept 14	Th	**Exam I**						
4	Sept 21	Th	Intro Spectros	IR	UV-Vis				
5	Sept 26	T	Molecular Spectroscopy	UV-Vis Experiment 13 Carla	IR Experiment 17 Audrius				
	Sept 28	Th							
6	Oct 3	T	Fluorescence	Fluorescence Experiment 15 Carla	FAA Experiment 12 Jorge				
	Oct 5	Th	AA						
7	Oct 10	T	AA/Vibrational	FAA Experiment 12 Jorge	Fluorescence Experiment 15 Carla				
	Oct 12	Th	Vibrational **Take Home Exam II**						
8	Oct 17	T	Mid Semester Break	Mid semester Break	Mid semester Break				
	Oct 19	Th	NMR						
9	Oct 24	T	NMR	NMR Experiment 18 Audrius	ASV Experiment 7 Jorge				
	Oct 26	Th	**Orozco: Demo Sampling**						

Oct 27	F	MidTerm Grades Are Due				

10	Oct 31	T	MS	ASV Experiment 7 Jorge	NMR Experiment 18 Audrius				
	Nov 2	Th	**Orozco: LUC Field Trip**						
11	Nov 6	M	Last Day to Withdraw						
	Nov 7	T	Intro to Electro	Sample Prep Experiment 23 Rotation	Sample Prep Experiment 23 Rotation				
	Nov 9	Th	**Orozco: Public Health**						
12	Nov 14	T	Potentiometry	Sample Analysis: Student Decision: Based on Linear Range, LOD, Time, Reliability					
	Nov 16	Th	**Orozco: Return Data** **Assist Graphing**						
13	Nov 21	T	ASV/ **Take Home Exam III**	ICP-MS Experiment 19 Luke	GC Experiment 22 Audrius				
	Nov 23	Th	Thanksgiving						
14	Nov 28	T	Separations	GC Experiment 22 Audrius	ICP-MS Experiment 19 Luke				
	Nov 30	Th	Separations						
15	Dec 5	T	Separations	Ethics & How do we Know the “Truth”					

311 Lab

Text: Lead Lab

Materials required: lab book with carbon pages, 3.5" diskettes

Group work:

Students will work in groups of nor more than 3 and no more than two groups per lab section. The group will be responsible for assigning work associated with the making of standards and the operation of each piece of equipment.

Group composition: Groups will be formed in the first week of lab. Students will interview each other for similarity of commutes and work habits in order to ensure that all members of a group can meet at some mutually agreeable time. Your best friend does not necessarily make your best lab partner in this context.

Scope of Work: Students **must** **pre-read** the lab. Sometimes work out of assigned class period is required. This will be compensated by cancelling lecture periods.

Each Student Will be monitored to ensure proper rotation of work.

The **Weekly** Reports

Labor division:

a) Data collection, tabulation, summarizing
b) Research
c) Editing and Copying

are three tasks associated with each report. Each person in the group **must** rotate tasks. Attach the following sheet to each lab (see next).

What the report looks like:

a) introduction
b) materials and methods
C. reduced data (graphs and tables). Graphs are to be inserted immediately after the first reference to the graph in the text. Graphs are to be labeled 1, 2, 3, etc. with a descriptive title.
d) **an essay that is not simply a list of answers to questions**
e) raw data

What you turn in:

a) The typed report
b) Work Distribution Form, with division of labor signed by each student

c) Carbon copy from page of labbook from each student
d) Individual data as relevant

When you turn it in:

Next lab
Provisional grade given
Edited and redone material with the original submission containing faculty commentary must be re-turned in 1 week after faculty edits.
Two rewrites only are allowed.

What if my group drives me nuts?

The groups can/will be configured three weeks into the semester. Be frank with your colleagues about their effort. If the group doesn't think you are pulling your weight you will end up doing **ALL** the work yourself!!

Lab		Student: ________	Student: ________	Student: ________
Potatos	Data/Graphs			
	Writing/Typing			
	Research/Editing			
ADC	Data/Graphs			
	Writing/Typing			
	Research/Editing			
$Pb(OH)_2$	Data/Graphs			
	Writing/Typing			
	Research/Editing			
Field Sampling	Data/Graphs			
	Writing/Typing			
	Research/Editing			
Sample Preparation				
	Data/Graphs			
	Writing/Typing			
	Research/Editing			
GFAA	Data/Graphs			
	Writing/Typing			
	Research/Editing			
Molecular Absorbance				
	Data/Graphs			
	Writing/Typing			
	Research/Editing			
IR	Data/Graphs			
	Writing/Typing			
	Research/Editing			
NMR	Data/Graphs			
	Writing/Typing			
	Research/Editing			
Mass Spec	Data/Graphs			
	Writing/Typing			
	Research/Editing			
ASV	Data/Graphs			
	Writing/Typing			
	Research/Editing			
CV	Data/Graphs			
	Writing/Typing			
	Research/Editing			
HPLC	Data/Graphs			
	Writing/Typing			
	Research/Editing			
GC	Data/Graphs			
	Writing/Typing			
	Research/Editing			

This page should be inserted with each lab report:

Method	Limit of Detection ug Pb/g solution	Linear Range ugPb/g	Interfences	Ease of Use: Solvent and/or Toxic Materials Used
Gravimetric (Chromate)				
Ion Selective Electrode				
Dithizone				
Calcein Blue				
ASV				
FAA				
GFAA				
ICP-MS				
IR				
NMR				
GC				