Chemistry 310 Instrumental Analysis "Lecture"
Spring 2012

This lab, although given separate credit and separate title from Chemistry 310, is completely integrated to Chemistry 310. It is not possible to take Chemistry 310 without performing the laboratory exercises 311.

The "lecture" section is designed to move along as closely as possible with the work in the lab. Time is allotted in each lecture section to discuss concepts and data obtained within the lab, as a result each student is expected to come to class prepared to ask questions and discuss the material from lab.

Grading

Best 2 of 3 exams	200	
Final	100	
GC-MS self guided	100	A group effort is required for the GC module. This module consists of questions and answers.
Participation	**40**	
Total	440 points	

There is NO ROUNDING at the end. Grades are assigned by

Total points		440
A	0.9	396
B	0.8	352
C	0.7	308
D	0.6	264

Grades of + and – are assigned at the discretion of the instructor.

The final consists of a poster presentation by each individual student discussing

- Their data for standard lead analysis on the various instruments and the data with respect to their dust wipe.
- A complete analytical proposal for the analysis of a sample/target analyte of their choice. The validity of each step of the analysis must be justified.

The grade of the poster is determined by a panel of judges using a standard rubric.

The criteria for "B" of the poster.

Each individual is expected to identify some topic of interest for analysis in addition to the dust wipes they have obtained. That analysis will typically be lead in some material, but may include some other method (such as GC-MS).

The individual is to decide upon a method of analysis based upon a consideration of the limits of detection of the instrument with respect to the public health limits associated with the material to be analyzed.

As an example: suppose the EPA determines that soils containing lead above 400 ppm can cause an increase in the blood lead level of a child and must be remediated. The method decided upon by the individual involves sampling 1 g of soil, digesting 0.25 g of the soil, collecting the digestate into a 50 mL volumetric. During the analysis 5 mL of the digestate were brought to a 100 mL volume. The instrumental limits for this condition will be 0.24 ppm

$$LOD_{instrument} << \left(400\,ppm_{action\ level}\right)\frac{\left(\frac{10^{-6}\,g_{Pb}}{1g_{soil}}\right)}{ppm}\frac{0.24g_{soil}}{50mL_{digestate}}\left(\frac{5mL_{digestate}}{40mL_{analysis}}\right) \tag{0.1}$$

$$LOD_{instrument} << \frac{400x10^{-6}x0.24g_{Pb}x5}{50x40mL_{analysis}} = \frac{0.24x10^{-6}\,g_{Pb}}{1ml_{analysis}} = 0.24\,ppm$$

The instrument chosen must be able to make measurements below the value of 0.24 ppm. If not then a zero reading on the instrument could be obtained even when a sample has a final 0.24 ppm diluted value, leading to the conclusion that the soil would not have to be remediated.

The individual must demonstrate that each step of the method is accurate and contributes no error to the method. In the example above the student needs to demonstrate a method which ensures that
a) the solvent used to digest the sample did not ADD lead to the digestate.
b) the collection of the digestate into a 50 mL volumetric did not LOSE lead from the sample.
c) the method chosen for digestion does indeed quantitatively transfer a known amount of lead from the soil into the digestate.
Etc.

I. Poster Evaluation (1 pt each)

For this section see the following web site:
http://www.makesigns.com/SciPosters_Home.aspx
For poster templates and hints about preparing scientific posters

Presenter has spent enough time to

1. Write in scientific English
2. Spell Checked
3. Punctuation and style is appropriate.

Is the poster readable?

4. Title is visible easily
5. Sections are clearly marked and titles are easy to read
6. Font size is appropriate for reading

Is the poster content arranged well?

7. Sections flow in a logical fashion for the content
8. Graphics are placed in a logical place for the text content
9. Graphics add to the visual spacing of the poster and do not detract

Does the poster have the parts appropriate for a scientific presentation?

10. Title
11. Purpose/Hypothesis
12. Sampling/Procedures
13. Results/Conclusions
14. Cited Literature

Is the data presented in an understandable format?

15. Graphs have a title and number and are referred to properly in the text
16. Axis are labeled correctly
17. Font size on the Axis and Caption are readable
18. Units are present in the axis labels
19. Graphs have a caption
20. Tables are easy to read
21. Tables have decent column headings
22. Tables have Title and Number and are referred to properly in the text.

II. Project Evaluation (4 pt each)

The project purpose/definition

1. The project has a clearly defined comparison to be tested
2. The comparison to be tested has been literature searched so that expected differences/values/action trigger levels, if available, are presented.

Sampling

- The samples proposed for collection are adequate for the question proposed and the expected action limits
- The splitting of samples is sufficient to test solvents and spikes and test for the precision necessary to determine if the sample concentration is above the Limit of Detection.

Instrumentation

- The project instrumental methodology is appropriate
- The methodology is a validated method?
- If not the method selected has a defendable rationale

- The presenter has shown that the instrument works well in previous work or literature.
- The calibration curve is appropriate for the expected sample concentration.
- The calibration curve is shown to be appropriate for the sample matrix after preparation.
- The LOD and LR of the instrument selected is appropriate for the expected values or action level values for the type of sample collected.

Solvents and Blanks

- The presenter shows that the solvents and reagents will not contribute lead content.
- The presenter shows that the solvents and reagents will not affect the quality of the calibration curve.

Accuracy

- The presenter showed that he/she is capable of carrying a sample through preparation and to instrumental analysis accurately by use of a spiked sample
- The presenter has identified a certified reference material and demonstrate that he/she obtained data consistent with the certified values

OTHER –free point for the evaluator based on their subjective response to the poster.

Chemistry 311 Instrumental Analysis Laboratory
Spring 2012

This lab, although given separate credit and separate title from Chemistry 310, is completely integrated to Chemistry 310. It is not possible to take Chemistry 310 without performing the laboratory exercises 311.

Responsibility of Students for Preparation and Cleanliness

There are four official lab times (M and F afternoons, T and W mornings). Each lab is 4 hours long. Students are expected to arrive with a working knowledge of the content of the assigned lab and be ready to begin promptly in order to complete the various tasks. *T.A.s will check the lab book to determine that each student has written a synopsis of the work to be accomplished.*

Grades can drop if laboratory cleanliness is not adhered to. Each group is responsible for the cleaning of all lab ware used and to return the equipment to the appropriate space. If this becomes an issue the groups, semester grade may be lowered by a full grade.

Students must use a lab book carbon paper.

Groupings and Schedule

In order to allow each student hands on access to the equipment each lab is split into 2 to 3 groups, each group having no more than 3 participants. The groups will follow DIFFERENT schedules throughout the semester as indicated on the next page.

2 labs deal with manipulation of data.

Working in groups is not easy. We expect you to make an honest effort to evaluate your own contribution and that of your partners to the group. At week three you will be given an opportunity to restructure. If an individual performs so poorly within a group that they are not "desirable" they will be expected to complete the work on their own with no decrease in the amount of work.

Points and Grades:

There are 11 labs. A formal lab report by the group is required for 6: Statistics, Electronics, UV-Vis, IC, ISE, and ASV. One group lab grade is dropped, for a total of 5 grades. An individual effort, of a differing format (rubric included below) is required for 3: AA, F, and IR. The net total possible points are 800. There is no rounding of grades:

Total points		800
A	0.9	720
B	0.8	640
C	0.7	560
D	0.6	480

Grades of – and + MAY be assigned at the discretion of the instructor in consultation with the TAs. Lack of cleanliness can result in a full grade drop.

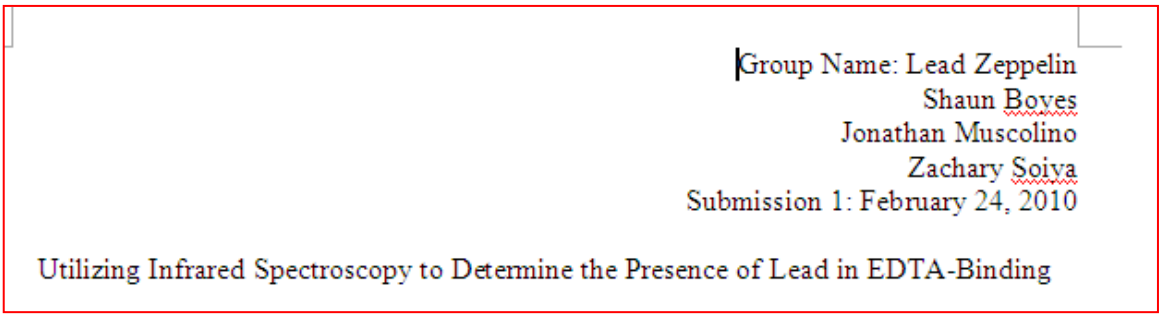
Group LAB REPORT GRADING

Lab reports generally run 10-15 pages.
They are submitted electronically, 1 week after the lab was completed.
You will receive a marked and edited copy of the lab 1 week after submission.
You have 1 week to either
a) respond to the written comments and return the lab for a higher grade
Or
b) accept the preliminary grade.
It goes without saying that I expect the papers submitted to be spell checked.
This process applies to all labs.

Each lab should contain the following sections:

A. A descriptive title

Notice that this document contains the group name, an indication that it is the first submission, the date of that first submission, and a title. When submitted electronically the version number should be indicated. Thus the **electronic file name** for this would be


Group Name: Lead Zeppelin
Shaun Boyes
Jonathan Muscolino
Zachary Soiya
Submission 1: February 24, 2010

Utilizing Infrared Spectroscopy to Determine the Presence of Lead in EDTA-Binding

should be : **Boy Mus Soi IR 02 24 version 1**

B. Introduction/Purpose

C. Short Materials/Methods (DO NOT COPY AND PASTE METHODS FROM THE INSTRUCTIONS) section rewritten by the students to reflect their knowledge of the methods. You may wish to use what you write for your lab book for entrance into the lab (see above under responsibilities).

C. Data AND Discussion combined.

Data here refers to analyzed data in the form of Tables and Graphs.

Within the discussion the group should meditate on the questions in each lab. The questions have been written to trigger some association between the exercises performed in lab and the concepts explored in both lab and "lecture". Consequently it is anticipated that the questions serve as a spring board to writing.

Writing a list of answers is NOT ACCEPTABLE. The data acquired within the lab should be used to illustrate important concepts identified by the reading and discussion of the students. You should consider this section to be a story telling section.

What is the story of this lab?
Why is it an interesting story?

What are the elements of the exercises in the lab that are essential to the story telling process?

For labs in which lead is the analyte **YOU MUST submit an LOD table** as part of your discussion section which provides a concentration based limit of detection determined by your group for the current lab and ALL preceding labs. You will discuss the differences between the current lab and ALL preceding labs as part of section C.

D. Appendix (Raw data as necessary)

Separate submission individually: For the first 3 weeks, you should send in at the same time as the lab report is submitted an individual evaluation of the type and quality of work performed by your other team members and of yourself.

FORMATING

1. Each graph should contain a labeled X and Y axis.
2. The font size in excel before import into your document should be bold, and at a minimum, 14 font.
2. The legend for any graph or table should be attached to the graph/table – No widows/orphans. A widow and orphan is a title that occurs on one page with the graph following on the second.
3. The graphs and figures should have a descriptive title and be numbered sequentially.
4. The graph location within the document follows immediately from the first discussion of that graph or figure.
5. Do not rotate the graphs. Keep them aligned with the document for ease of reading.

Grading of the three individual lab reports

Table 2. Assessment Rubric for Solutions to Laboratory Problems (39 points total)[1]

Section	Criteria	Description & Characteristics
Introduction	Context	Report demonstrates a clear understanding of the 'big picture' and addresses the following questions: • Why is this question important/useful/ necessary in chemical analysis? • What do we know already? What problem/question is this experiment addressing?
	Accuracy & Relevance	Content knowledge described relevant to this experiment is accurate, relevant, and provides appropriate background information, including defining critical terms.
Questions & Hypothesis/es	Testable	Hypothesis/es and/or questions are clearly stated, testable, scientifically relevant and consider plausible alternative explanations where necessary.
Methods	Controls & Replication	Appropriate controls (including appropriate replication) are present and explained.
	Experimental Design	Experimental design is likely to produce salient and fruitful results. The design focuses on relevant tests for the hypothesis/es & question(s) posed.
Results	Data Selection	Data chosen are comprehensive, accurate, and relevant.
	Data analysis	Data analysis is appropriate for hypotheses tested and appears correctly performed and interpreted with relevant values reported and explained.

[1] derived from Timmerman, et. al 2011

Section	Criteria	Description & Characteristics
	Data presentation	Data are summarized in logical format. Table or graph types are appropriate. Data are properly labeled including units. Graphs are appropriately labeled and scaled. Captions, if any, are informative and complete.
Discussion	Conclusions	Conclusions are clearly and logically drawn from data provided. A logical chain of reasoning from hypothesis to data to conclusions is clearly and persuasively explained. Conflicting data, if present, are adequately addressed.
	Alternative Explanations	Alternative explanations (hypotheses) are considered and clearly eliminated in persuasive discussion.
	Limitations of design	Limitations of the data and/or experimental design and corresponding implications for data interpretation are discussed.
Connection to other knowledge		Writer provides a relevant, accurate, and reasonable discussion of how this experiment relates to other knowledge in the chemistry.
Writing quality		Grammar, word usage and organization facilitate understanding of the report.

Special Notes	Week #	Date	Proposed Experiment		
			Schedule 1	Schedule 2	Schedule 3
MLK DAY		Monday, January 16, 2012	----	----	----
	1	Tuesday, January 17, 2012	Lab Intro, expectations, etc.	Lab Intro, expectations, etc.	Lab Intro, expectations, etc.
		Wednesday, January 18, 2012	Lab Intro, expectations, etc.	Lab Intro, expectations, etc.	Lab Intro, expectations, etc.
		Friday, January 20, 2012	Lab Intro, expectations, etc.	Lab Intro, expectations, etc.	Lab Intro, expectations, etc.
		Monday, January 23, 2012	Lab Intro, expectations, etc.	Lab Intro, expectations, etc.	Lab Intro, expectations, etc.
	2	Tuesday, January 24, 2012	Statistics	Statistics	Statistics
		Wednesday, January 25, 2012	Statistics	Statistics	Statistics
		Friday, January 27, 2012	Statistics	Statistics	Statistics
		Monday, January 30, 2012	Statistics	Statistics	Statistics
	3	Tuesday, January 31, 2012	Electronics (Matthew)	Electronics (Matthew)	Electronics (Matthew)
		Wednesday, February 01, 2012	Electronics (Matthew)	Electronics (Matthew)	Electronics (Matthew)
		Friday, February 03, 2012	Electronics (Matthew)	Electronics (Matthew)	Electronics (Matthew)
		Monday, February 06, 2012	Electronics (Matthew)	Electronics (Matthew)	Electronics (Matthew)
	4	Tuesday, February 07, 2012	Digestions	Digestions	Digestions
		Wednesday, February 08, 2012	Digestions	Digestions	Digestions
		Friday, February 10, 2012	Digestions	Digestions	Digestions
		Monday, February 13, 2012	Digestions	Digestions	Digestions
	5	Tuesday, February 14, 2012	IR (Mary)	IR (Mary)	UV-Vis (Jonathan)
		Wednesday, February 15, 2012	IR (Mary)	IR (Mary)	UV-Vis (Jonathan)
		Friday, February 17, 2012	IR (Mary)	IR (Mary)	UV-Vis (Jonathan)
		Monday, February 20, 2012	IR (Mary)	IR (Mary)	UV-Vis (Jonathan)
	6	Tuesday, February 21, 2012	UV-Vis (Jonathan)	UV-Vis (Jonathan)	IR (Mary)
		Wednesday, February 22, 2012	UV-Vis (Jonathan)	UV-Vis (Jonathan)	IR (Mary)
		Friday, February 24, 2012	UV-Vis (Jonathan)	UV-Vis (Jonathan)	IR (Mary)
		Monday, February 27, 2012	UV-Vis (Jonathan)	UV-Vis (Jonathan)	IR (Mary)
	7	Tuesday, February 28, 2012	AA (Matthew)	GC module (Dr. Fitch)	Fluorescence (Jonathan)
		Wednesday, February 29, 2012	AA (Matthew)	GC module (Dr. Fitch)	Fluorescence (Jonathan)
		Friday, March 02, 2012	AA (Matthew)	GC module (Dr. Fitch)	Fluorescence (Jonathan)
BREAK -- NO CLASS		Monday, March 05, 2012	----	----	----
BREAK -- NC	8	Tuesday, March 06, 2012	----	----	----
BREAK -- NO CLASS		Wednesday, March 07, 2012	----	----	----
BREAK -- NO CLASS		Friday, March 09, 2012	----	----	----
		Monday, March 12, 2012	AA (Matthew)		Fluorescence (Jonathan)
	9	Tuesday, March 13, 2012	Fluorescence (Jonathan)	AA (Matthew)	GC module (Dr. Fitch)
		Wednesday, March 14, 2012	Fluorescence (Jonathan)	AA (Matthew)	GC module (Dr. Fitch)
		Friday, March 16, 2012	Fluorescence (Jonathan)	AA (Matthew)	GC module (Dr. Fitch)
		Monday, March 19, 2012	Fluorescence (Jonathan)	AA (Matthew)	GC module (Dr. Fitch)
	10	Tuesday, March 20, 2012	GC module (Dr. Fitch)	Fluorescence (Jonathan)	AA (Matthew)
		Wednesday, March 21, 2012	GC module (Dr. Fitch)	Fluorescence (Jonathan)	AA (Matthew)
		Friday, March 23, 2012	GC module (Dr. Fitch)	Fluorescence (Jonathan)	AA (Matthew)
		Monday, March 26, 2012	GC module (Dr. Fitch)	Fluorescence (Jonathan)	AA (Matthew)
	11	Tuesday, March 27, 2012	----	----	----
		Wednesday, March 28, 2012	----	----	----
		Friday, March 30, 2012	ISE (Mary)	IC (Matthew)	ASV (Jonathan)
		Monday, April 02, 2012	ISE (Mary)	IC (Matthew)	ASV (Jonathan)
	12	Tuesday, April 03, 2012	ISE (Mary)	IC (Matthew)	ASV (Jonathan)
		Wednesday, April 04, 2012	ISE (Mary)	IC (Matthew)	ASV (Jonathan)
CLASSES -- EASTER		Friday, April 06, 2012	----	----	----
CLASSES -- EASTER		Monday, April 09, 2012	----	----	----
	13	Tuesday, April 10, 2012	ASV (Jonathan)	ISE (Mary)	IC (Matthew)
		Wednesday, April 11, 2012	ASV (Jonathan)	ISE (Mary)	IC (Matthew)
		Friday, April 13, 2012	ASV (Jonathan)	ISE (Mary)	IC (Matthew)
		Monday, April 16, 2012	ASV (Jonathan)	ISE (Mary)	IC (Matthew)
	14	Tuesday, April 17, 2012	IC (Matthew)	ASV (Jonathan)	ISE (Mary)
		Wednesday, April 18, 2012	IC (Matthew)	ASV (Jonathan)	ISE (Mary)
		Friday, April 20, 2012	IC (Matthew)	ASV (Jonathan)	ISE (Mary)
		Monday, April 23, 2012	IC (Matthew)	ASV (Jonathan)	ISE (Mary)
	15	Tuesday, April 24, 2012	take final exam	take final exam	take final exam
		Wednesday, April 25, 2012	take final exam	take final exam	take final exam
		Friday, April 27, 2012	take final exam	take final exam	take final exam
		Monday, April 30, 2012	take final exam	take final exam	take final exam

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Alanah's Schedule Spring 2012

	Mon	Tues	Wed	Thur	Fri
8:30		Lab 02W	Lab 03W		
9:20					Group meeting 11-12-11
10:25	T.A. Meeting				
11:30					
12:20	Office Hour	Office Hour			Office Hour
1:40-2:30	Class DU-4		Class Du-4		Class DU-4
2:45	Lab 01W				
3:45			Faculty Council Once/month	Seminar 4-5:15 LSB 145	Lab 04W
4:55					
6:00					
6:35					
7:35					

Alanah's Travel Schedule:

Mar 1-2 National Science Foundation Panel, Washington D.C.
Mar 9-11 Pittcon Orlando, Analytical Sciences Digital Library Executive Committee
Apr 7-11 Electrochemical Society Meeting Seattle