Chemistry 311 Spring 2011

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. 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.

## **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:**

9 labs each with a written lab report plus 1 three week project culminating in a group poster. One lab grade is dropped for a total of 900 points.

There is no rounding of grades:

0.9	810 A
8.0	720 B
0.7	630 C
0.6	540 D
0.5	450 F

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.

Week#	Date	Proposed Experiment			Project Schedule
		Schedule 1	Schedule 2	Schedule 3	
	Monday, January 17, 2011				
1	Tuesday, January 18, 2011	Statistics	Statistics	Statistics	Projects Explained in Lab
	Wednesday, January 19, 2011	Statistics	Statistics	Statistics	Projects Explained in Lab
	Friday, January 21, 2011	Statistics	Statistics	Statistics	Projects Explained in Lab
	Monday, January 24, 2011	Statistics	Statistics	Statistics	Projects Explained in Lab
2	Tuesday, January 25, 2011	Electronics (Matthew)	Electronics (Matthew)	Electronics (Matthew)	
	Wednesday, January 26, 2011	Electronics (Matthew)	Electronics (Matthew)	Electronics (Matthew)	
	Friday, January 28, 2011	Electronics (Matthew)	Electronics (Matthew)	Electronics (Matthew)	
	Monday, January 31, 2011	Electronics (Matthew)	Electronics (Matthew)	Electronics (Matthew)	
3	Tuesday, February 01, 2011	ISE (Mary)	UV-Vis (Maria)	UV-Vis (Maria)	Project Proposal Due
	Wednesday, February 02, 2011	ISE (Mary)	UV-Vis (Maria)	UV-Vis (Maria)	Project Proposal Due
	Friday, February 04, 2011	ISE (Mary)	UV-Vis (Maria)	UV-Vis (Maria)	Project Proposal Due
	Monday, February 07, 2011	ISE (Mary)	UV-Vis (Maria)	UV-Vis (Maria)	Project Proposal Due
4	Tuesday, February 08, 2011	UV-Vis (Maria)	ISE (Mary)	ISE (Mary)	Troject Toposar Bac
4	Wednesday, February 09, 2011	UV-Vis (Maria)	ISE (Mary)	ISE (Mary)	
	Friday, February 11, 2011	UV-Vis (Maria)	ISE (Mary)	ISE (Mary)	
		. ,			Conference Schedule with Dr. Fitch and/or TAs?
-	Monday, February 14, 2011	UV-Vis (Maria)	ISE (Mary)	ISE (Mary)	Conference Schedule with Dr. Fitch and/or TAS:
5	Tuesday, February 15, 2011	IR (Mary)	AA (Matthew)	ASV (Maria)	·
	Wednesday, February 16, 2011	IR (Mary)	AA (Matthew)	ASV (Maria)	Conference Schedule with Dr. Fitch and/or TAS?
	Friday, February 18, 2011	IR (Mary)	AA (Matthew)	ASV (Maria)	Conference Schedule with Dr. Fitch and/or TAs?
	Monday, February 21, 2011	IR (Mary)	AA (Matthew)	ASV (Maria)	Methods Due for all Lab Sections
6	Tuesday, February 22, 2011	ASV (Maria)	IR (Mary)	AA (Matthew)	
	Wednesday, February 23, 2011	ASV (Maria)	IR (Mary)	AA (Matthew)	
	Friday, February 25, 2011	ASV (Maria)	IR (Mary)	AA (Matthew)	
	Monday, February 28, 2011	ASV (Maria)	IR (Mary)	AA (Matthew)	
7	Tuesday, March 01, 2011	AA (Matthew)	ASV (Maria)	IR (Mary)	
	Wednesday, March 02, 2011	AA (Matthew)	ASV (Maria)	IR (Mary)	Materials Due for all Lab Sections
	Friday, March 04, 2011	AA (Matthew)	ASV (Maria)	IR (Mary)	
	Monday, March 07, 2011				
8	Tuesday, March 08, 2011				
	Wednesday, March 09, 2011				
	Friday, March 11, 2011				
	Monday, March 14, 2011	AA (Matthew)	ASV (Maria)	IR (Mary)	
9	Tuesday, March 15, 2011	NMR (Mary)	GC/MS (Matthew)	Fluorescence (Maria)	
	Wednesday, March 16, 2011	NMR (Mary)	GC/MS (Matthew)	Fluorescence (Maria)	
	Friday, March 18, 2011	NMR (Mary)	GC/MS (Matthew)	Fluorescence (Maria)	
	Monday, March 21, 2011	NMR (Mary)	GC/MS (Matthew)	Fluorescence (Maria)	
10	Tuesday, March 22, 2011	Fluorescence (Maria)	NMR (Mary)	GC/MS (Matthew)	
10	Wednesday, March 23, 2011	Fluorescence (Maria)	NMR (Mary)	GC/MS (Matthew)	
	Friday, March 25, 2011	Fluorescence (Maria)	NMR (Mary)	GC/MS (Matthew)	
	Monday, March 28, 2011	Fluorescence (Maria)	NMR (Mary)	GC/MS (Matthew)	
11					
11	Tuesday, March 29, 2011	GC/MS (Matthew)	Fluorescence (Maria) Fluorescence (Maria)	NMR (Mary) NMR (Mary)	
	Wednesday, March 30, 2011	GC/MS (Matthew)			
	Friday, April 01, 2011	GC/MS (Matthew)	Fluorescence (Maria)	NMR (Mary)	
	Monday, April 04, 2011	GC/MS (Matthew)	Fluorescence (Maria)	NMR (Mary)	
12	Tuesday, April 05, 2011	Projects (Digestions/Analysis)	Projects (Digestions/Analysis)	Projects (Digestions/Analysis)	
	Wednesday, April 06, 2011	Projects (Digestions/Analysis)	Projects (Digestions/Analysis)	Projects (Digestions/Analysis)	
	Friday, April 08, 2011	Projects (Digestions/Analysis)	Projects (Digestions/Analysis)	Projects (Digestions/Analysis)	
	Monday, April 11, 2011	Projects (Digestions/Analysis)	Projects (Digestions/Analysis)	Projects (Digestions/Analysis)	
13	Tuesday, April 12, 2011	Projects (Sample/Data Analysis)	Projects (Sample/Data Analysis)	Projects (Sample/Data Analysis)	
	Wednesday, April 13, 2011	Projects (Sample/Data Analysis)	Projects (Sample/Data Analysis)	Projects (Sample/Data Analysis)	
	Friday, April 15, 2011	Projects (Sample/Data Analysis)	Projects (Sample/Data Analysis)	Projects (Sample/Data Analysis)	
	Monday, April 18, 2011	Projects (Sample/Data Analysis)	Projects (Sample/Data Analysis)	Projects (Sample/Data Analysis)	
14	Tuesday, April 19, 2011	Projects (Clean-up and Misc.)	Projects (Clean-up and Misc.)	Projects (Clean-up and Misc.)	
	Wednesday, April 20, 2011	Projects (Clean-up and Misc.)	Projects (Clean-up and Misc.)	Projects (Clean-up and Misc.)	
	Friday, April 22, 2011				
	Monday, April 25, 2011				
15	Tuesday, April 26, 2011	Projects (Clean-up and Misc.)	Projects (Clean-up and Misc.)	Projects (Clean-up and Misc.)	
	Wednesday, April 27, 2011	Projects (Clean-up and Misc.)	Projects (Clean-up and Misc.)	Projects (Clean-up and Misc.)	
	Friday, April 29, 2011	Projects (Clean-up and Misc.)	Projects (Clean-up and Misc.)	Projects (Clean-up and Misc.)	

## The 9 lab reports (8 counting toward your grade)

7 of the 10 labs (Ion Selective Electrodes, UV-Vis, IR, Anodic stripping voltammetry, Atomic Absorption, NMR, and fluorescence) measure either the quantitative amount of lead present in a sample, or determine the structural chemistry of a lead chelate.

#### 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. How the lab reports are graded. It goes without saying that I expect the papers 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

might be: Zeppelin IR 02 24 version 1

## **B.** Introduction/Purpose

<u>C. Short Materials/Methods</u> (DO NOT COPY AND PASTE METHODS FROM THE INSTRUCTIONS) section rewritten by the student to reflect their knowledge of the methods.

## 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 the are essential to the story telling process?

For 7/9 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)

<u>Separate submission individually:</u> 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.

## **Projects and Poster**

The final 3 weeks of the semester are grouped together as "1" lab. That "lab" is a group driven project. The group will be the same one you have been partnered with throughout the semester. This "lab" has as its report a poster. The criteria for grading of the poster is attached.

Each group is expected to identify some topic of interest for analysis. That analysis will typically be lead in some material, but may include some other method (such as GC-MS).

The group 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 group 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

$$\left[\frac{(400 ppm)\left(\frac{10^{-6} g_{pb}}{1g_{soil}}\right)(0.24g_{soil})}{50mL_{digestate}}\right] \left(\frac{5mL_{digestate}}{40mL_{analysis}}\right)$$

$$= \frac{5x0.24x400x10^{-6} g_{pb}}{50x40g_{soil}} = \frac{0.24x10^{-6} g_{pb}}{1g_{soil}} = 0.24ppm$$
The part he able to make measurements below the value of 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 group must demonstrate that each step of the method is accurate and contributes no error to the method. In the example above the students would need to demonstrate

- 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.

Students are expected to complete the work during their regularly scheduled lab periods.

How you will be tested on your Poster:

## 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 collected were

3. adequate for the question proposed and the expected action limits

4. The splitting of samples was 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

- 5. The methodology is a validated method?
- 6. If not the method selected has a defendable rationale
- 7. The presenter has shown that the instrument works well at the time that the data was collected
- 8. The calibration curve was appropriate for the expected sample concentration.
- 9. The calibration curve was shown to be appropriate for the sample matrix after preparation.
- 10. The LOD and LR of the instrument selected was appropriate for the expected values or action level values for the type of sample collected.
- 11. The experimental LOD obtained by the student for the calibration curve was compared in the text to the expected LOD obtained by that instrument in the literature for that sample.

#### Solvents and Blanks

- 12. The presenter showed that the solvents and reagents did not contribute lead content.
- 13. The presenter showed that the solvents and reagents did not affect the quality of the calibration curve.

#### Accuracy

- 14. The presenter showed that he/she was capable of carrying a sample through preparation and to instrumental analysis accurately by use of a spiked sample
- 15. The presenter was able to obtain a certified reference material and demonstrate that he/she obtained data consistent with the certified values

#### Conclusions

- 16. The conclusions drawn by the difference between the compared samples are appropriate.
- 17. The presenter explicitly compares the values obtained to the LOD they obtained experimentally, the expected LOD, and the action level for that type of sample.

OTHER – 10 free point for the evaluator based on their subjective response to the poster.

Total: 22+17\*4+10=100