Chemistry 310/311 Instrumental Analysis
Spring 2014/Alanah Fitch
Afitch@luc.edu
773-508-3119
Flanner Hall Room 402 (RING THE BELL!!!)

	8:30-12:20 Lab			
		10-12 Office Hours		
1:40-2:30 Cuneo		1:40-2:30 Cuneo		1:40-2:30 Cuneo
3-5 Office Hours	2:45-6:30 Lab		1-4:50 Lab	

Optional Text: **Analytical Sciences Digital Library**
http://home.asdlib.org/

Readings are provided the week before the material. Readings consist of articles and a summary document by the instructor.

The 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 in 311. In the following years the two classes will be "integrated" in the register's mind. For this year you will receive the same grade for both "lab" and "lecture".

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

Grade	%
A	90
B	80
C	70
D	60

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

Materials and Equipment to Be Supplied by the Student

Flash drive, laptop computer, pencils, calculator
Labbook, carbon tear out sheets

Responsibility of Students for Preparation and Cleanliness

There are three official lab times. 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. *A synopsis of a beginning question and workto be accomplished is due the night before the lab.*

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 below.

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.

ClassRoom Exercises Due at end of class	**Pts**	**Labs Due one week from completion**	**Pts**	**Description of Work**
		Statistics	50	Group Lab Report: Must be rewritten once addressing comments; if first draft <50% no rewrite allowed; grade: Fitch; regrade: Tas)
Calibration Curves and Confidence Limits	10			
		Data Filtering	50	Group Lab Report: Must be rewritten once addressing comments; if first draft <50% no rewrite allowed; grade: Fitch; regrade: Tas)
Cell Phone Spectrophotometer	10			Data submitted at end of "Lecture" class
Deconvolutions	10			Data submitted at end of "Lecture" class
Extractions	10			Data submitted at end of "Lecture" class
		UV-Vis	50	Group Lab Report: Must be rewritten once addressing comments; if first draft <50% no rewrite allowed; grade: Fitch; regrade: Tas)
IR FT transformations	10			Data submitted at end of "Lecture" class
		IR	50	Group Lab Report: Must be rewritten once addressing comments; if first draft <50% no rewrite allowed; grade: Fitch; regrade: Tas)
		Raman	50	Individual Executive Summary: includes cost instrumer description
Prep for IC: Selectivity and Masking	10			Data submitted at end of "Lecture" class
		IC 1	50	Individual Executive Summary:
		IC 2	wild card	TB decided based on first 3 lab report quality
modeling peak shapes	10			Data submitted at end of "Lecture" class

		ASV	50	Group Lab Report: Must be rewritten once addressing comments; if first draft <50% no rewrite allowed; grade: Fitch; regrade: Tas)
		AA	50	Individual Executive Summary: Write, and prepare to discuss in class, includes diagram, purpose of componen parts and costs
ASDL: GC-MS module	10			Data submitted at end of "Lecture" class
Temperature Programming	10			Data submitted at end of "Lecture" class
		GC	50	Individual Executive Summary: Write, and prepare to discuss in class, includes diagram, purpose of componen parts and costs
ASDL: Sampling module	10			Data submitted at end of "Lecture" class
		Sampling	50	Individual Executive Summary: Write, and prepare to discuss in class
		Digestions	50	Individual Executive Summary: Write, and prepare to discuss in class
		Data Analysis	50	Data analysis is due at END of LAB!!!!!!!
Tour ICP-MS				Individual Executive Summary: Write, and prepare to discuss in class, includes diagram, purpose of componen parts and costs
Class Final Research Proposals: Lake Nakuru: written	100			Individual complete proposal: hypothesis, sampling locations, sampling rationale, sample prep and rationale, and method, costs (estimated transportation and instrum and budget
Lab Final Poster: your work on site analysis			100	Includes all quality control and discusses individual data context of all data from this year and last year
Best 3 exams	300			
	500		700	Total: 1200 points
% distribution	41.7		58.3	

Lab Section	# Students		TA	Labs
Tuesday 8:30 - 12:20	7		Jonathan	Stats, Data Filtering, UV-Vis, Rama
Tuesday 2:45 - 6:30	9		Matthew	Stats, Data Filtering, IC, IR, AA, An
Thursday 1:00 - 4:50	8		Dr. Fitch	Stats, Data Filtering, GC, Sampling
Date	**Week #**	**Schedule 1**	**Schedule 2**	**Schedule 3**
Tuesday, January 14, 2014	1	Statistics (RM 007)	Statistics (RM 007)	Statistics (RM 007)
Thursday, January 16, 2014		Statistics (RM 007)	Statistics (RM 007)	Statistics (RM 007)
Tuesday, January 21, 2014	2	Data Filtering (RM 007)	Data Filtering (RM 007)	Data Filtering (RM 007)
Thursday, January 23, 2014		Data Filtering (RM 007)	Data Filtering (RM 007)	Data Filtering (RM 007)
Tuesday, January 28, 2014	3	UV-Vis (RM 002)	UV-Vis (RM 002)	UV-Vis (RM 002)
Thursday, January 30, 2014		UV-Vis (RM 002)	UV-Vis (RM 002)	UV-Vis (RM 002)
Tuesday, February 04, 2014	4	IR (RM 314)	Raman (RM 314)	IC Lab 1 (RM 314)
Thursday, February 06, 2014		IR (RM 314)	Raman (RM 314)	IC Lab 1 (RM 314)
Tuesday, February 11, 2014	5	Raman (RM 314)	IR (RM 314)	IC Lab 2 (RM 314)
Thursday, February 13, 2014		Raman (RM 314)	IR (RM 314)	IC Lab 2 (RM 314)
Tuesday, February 18, 2014	6	IC Lab 1 (RM 314)	ASV (RM 402)	IR (RM 314)
Thursday, February 20, 2014		IC Lab 1 (RM 314)	ASV (RM 402)	IR (RM 314)
Tuesday, February 25, 2014	7	IC Lab 2 (RM 314)	AA (RM 314)	Raman (RM 314)
Thursday, February 27, 2014		IC Lab 2 (RM 314)	AA (RM 314)	Raman (RM 314)
Tuesday, March 04, 2014	8	NO CLASS – SPRING BREAK	NO CLASS – SPRING BREAK	NO CLASS – SPRING BREAK
Thursday, March 06, 2014		NO CLASS – SPRING BREAK	NO CLASS – SPRING BREAK	NO CLASS – SPRING BREAK
Tuesday, March 11, 2014	9	ASV (RM 402)	GC (RM 314)	AA (RM 314)
Thursday, March 13, 2014		ASV (RM 402)	GC (RM 314)	AA (RM 314)
Tuesday, March 18, 2014	10	AA (RM 314)	IC Lab 1 (RM 314)	GC (RM 314)
Thursday, March 20, 2014		AA (RM 314)	IC Lab 1 (RM 314)	GC (RM 314)
Tuesday, March 25, 2014	11	GC (RM 314)	IC Lab 2 (RM 314)	ASV (RM 402)
Thursday, March 27, 2014		GC (RM 314)	IC Lab 2 (RM 314)	ASV (RM 402)
Tuesday, April 01, 2014	12	Sampling (FH Foyer)	Sampling (FH Foyer)	Sampling (FH Foyer)
Thursday, April 03, 2014		Sampling (FH Foyer)	Sampling (FH Foyer)	Sampling (FH Foyer)
Tuesday, April 08, 2014	13	Digestions (RM 402)	Digestions (RM 402)	Digestions (RM 402)
Thursday, April 10, 2014		Digestions (RM 402)	Digestions (RM 402)	Digestions (RM 402)
Tuesday, April 15, 2014	14	NO CLASS	NO CLASS	NO CLASS
Thursday, April 17, 2014		NO CLASS – EASTER	NO CLASS – EASTER	NO CLASS – EASTER
Tuesday, April 22, 2014	15	Data Analysis (TBD)	Data Analysis (TBD)	Data Analysis (TBD)
Thursday, April 24, 2014		Data Analysis (TBD)	Data Analysis (TBD)	Data Analysis (TBD)

A Writing Intensive Class

- Lab reports
- Scientific Articles
- Oral Presentations
- Poster Presentations
- Executive Summary Format
- Research Proposals

Your work will consist of a mixture of the above. The first part of the semester you will write formal lab reports that loosely mirror scientific articles in the quality of graphs and the writing style. You are expected to write a document of persuasion that the results you have obtained have a unique story to tell that is compelling and answers some question posed. These reports will be subjected to an editing process in which the writer MUST respond to the commentary.

Your second exam will tie your writing skills more specifically to scientific articles by having a take home section of commentary on a relevant scientific article related to lead analysis.

The second part of the semester you are assumed to have mastered the skill of graphing for publication. You will be asked to submit Executive Summaries.

The final project of the semester is a poster presentation which contains an executive summary of the class local project and your specific data quality. It also contains a research proposal which has been narrowly defined as a methodology to answer the question: "Why are/have flamingos died in Lake Nakuru, Kenya?"

Over the semester you will therefore have exposure to and practice at most of the major forms of scientific communication.

In three separate documents you can find specific instructions for the required lab formats for the first ½ of the semester; the format requested for the Executive Summary Format, and the format requested for the Poster Format.

FORMAT FOR AN EXECUTIVE SUMMARY

Executive Summary Format
Introductory Statement (not a section)
Write one or two sentences to introduce the report.
Objectives
State the objectives clearly and succinctly
Results
Present the results in easy-to-understand form. Graphs and tables are the most common forms of communicating results. (See Results in Lab Report format below.)
Conclusion
Give a clear, meaningful, statement of your findings.

http://www.me.uprm.edu/o_meza/Writing%20Lab%20Reports.htm#E

Consult also, the front page to any governmental technical report and look at the abstract:

Spot Test Kits For Detecting
Lead in Household Paint:
A Laboratory Evaluation

Walter J. Rossiter, Jr.*
Mark G. Vangel**
Mary E. McKnight*

ABSTRACT

A laboratory study was conducted to determine the reliability of spot test kits for detecting the presence of lead in household paint when tests were conducted by certified lead inspectors or risk assessors. Reagent solutions were applied to paint specimens and, subsequently, the specimens were observed for characteristic color change. For the study, four test kits were based on the reaction of lead ion with sulfide ion to produce a gray or black color, whereas four others were based on the reaction of lead ion with rhodizonate ion to give a pink or red color. These eight kits were used in an experiment investigating the effect of lead level, lead pigment type, operator, paint-film substrate, overlayer paint type, and overlayer paint thickness. Test samples, prepared using either a white lead (i.e., basic lead carbonate) or a lead chromate pigment, had ten lead levels ranging from 0 mg/cm2 to 3.5 mg/cm2. Five operators were trained according to test protocols based on each kit manufacturer's instructions. The study showed that the spot test kits gave positive results at lead levels less than 1 mg/cm2. Consequently, a positive response could not be relied on to indicate the presence of lead-based paint, which is defined as paint having lead levels equal to, or greater than, 1 mg/cm2. This finding is consistent with the results of past field studies. A criterion against which a spot test kit may be considered as acceptable for use as a negative screen (i.e., a test for which a negative result indicates a low probability of lead $ 1 mg/cm2) for the presence of lead-based paint was proposed. This criterion is: Upon evaluation of spot test kit response, the probability of a negative response (with 95 % confidence) at a lead level of 1 mg/cm2 is # 5%. Equivalently, the lead level at which there is a 95 % probability of a positive response (with 95 % confidence) should be # 1 mg/cm2. The type of lead pigment had a

significant effect on the spot test kit response. For white lead specimens, six kits—three sulfide-based and three rhodizonate-based—gave low percents of false negatives (# 2 %) and met the proposed criterion for acceptance as a negative screen for leadbased paint. For lead chromate specimens, three of these six kits—two sulfide-based and one rhodizonate-based—also had low percents of false negatives (# 2 %) and met the proposed acceptance criterion. The other factors—overlayer type, overlayer thickness, operator, and substrate—did not generally show significant effects in cases where the spot test kits appeared to be candidates for use as negative screens for lead-based paint. Finally, the study results lead to the suggestion that an evaluation of spot test kit response should afford a low percent of positive results at the 0 mg/cm2 lead level because, in practice, false-positives may needlessly spur test kit users intotaking further, but unnecessary, investigative action for the presence of lead.

Key words: building technology; detection; kit response; lead-based paint; lead level; lead chromate; operator effect; spot test kits; testing; white lead

LAB REPORT FORMAT

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. File Name for Electronic Submission

For a group of three students whose last names are Shah, Baker, Chang submitting a report on the IR lab on February 24 for the first time the electronic file name MUST BE

Bak Cha Sha IR 02 24 V1.doc

The names are listed last name by alphabet. You will get the document back labeled as
Bak Cha Sha IR 02 24 V1 Fitch commented.doc

For the resubmission 1 week later the document electronic name MUST BE

Bak Cha Sha IR 03 01 V2.doc

B. A descriptive title

Notice that this document contains the group name (not necessary), the submitters names in alphabetical order, an indication that it is the first submission, the date of that first submission, and a title.

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

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

C.Introduction/Purpose/your proposed question

Here you pose your question as outlined in the box below. Your Question is DUE on night before arrival in the lab. Submit electronically.

The Process of the Science Writing Heuristic

Beginning Questions

a. Propose a beginning question to explore the purpose for doing the experiment.

b. A beginning question should be of the form "How does one variable depend on another variable?"

c. Beginning questions that are not acceptable include:

   1. "Why?" questions.
   2. Factoid questions.
   3. Questions that can be answered without doing the experiment.

d. Can you make a prediction to try to answer your beginning question?

Procedure and Tests

a. Propose your plan for how the beginning questions can be answered by doing the experiment. (This may be different from what you actually *do* during the experiment, but it is a start.)

b. Make an outline of precisely what you did (after sharing ideas with your group and drafting a group strategy).

D. 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).

E. Data AND Discussion combined.

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

You may have written lab reports for other classes in which your data was presented and then the discussion. I require a different format. The format required is intended help you interpret your data. I want to see well made graphs/tables within the context of your discussion/interpretation of the data. As part of the discussion a few select articles have been provided so that you need not research the literature extensively to find the context for your data. I have also included some trigger words/questions to be discussed by your group in preparing the lab report.

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/point/question posed by you 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.

F. Individual Reflections For Each Student Come Before any appendices

Reflection (16 points total):

- Have I identified and explained sources of error and assumptions made during the experiment?
- How have my ideas changed, what new question do I have, what new things do I have to think about?
- How does this work tie to concepts about which I have learned in class?
- To what can I refer in my text, my notes, or some real-life application to make a connection with this laboratory work?

G. Appendix (Raw data as necessary)

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.

Examples of good and "bad" graphs follow

EXAMPLE 1

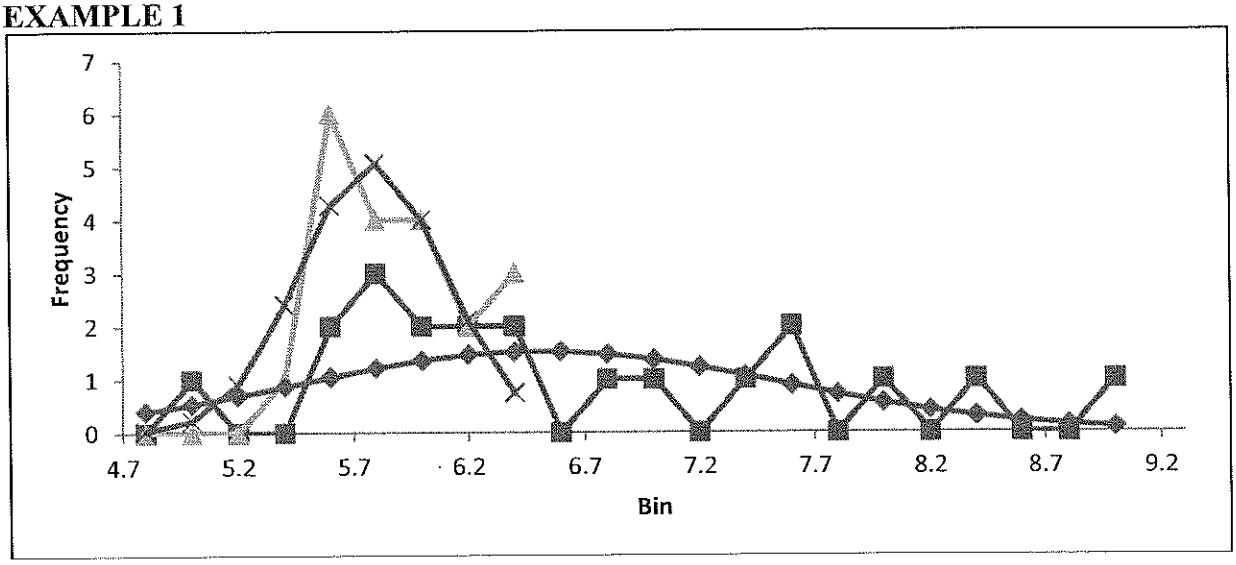


Figure 1: Still looking only at the major axis of the potato populations. Bin size to frequency for red and gold potato populations vs. their corresponding Gaussian. The green data represents the red potatoes bin to frequency values, based off their major axis measurements, and the red corresponds to white potatoes. The purple is the red potatoes corresponding Gaussian and the Blue curve corresponds to the white potatoes Gaussian.

We also made a scatter plot of the major axis bin to frequency with a Gaussian curve for the hypothesis that the potatoes came from one population (Figure 2).

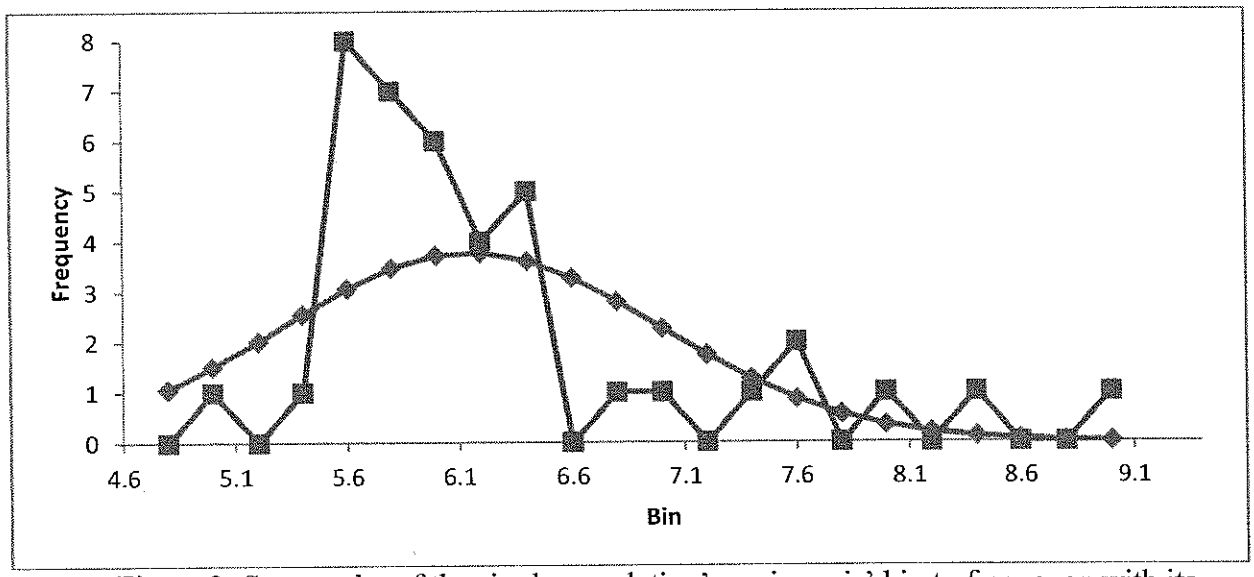
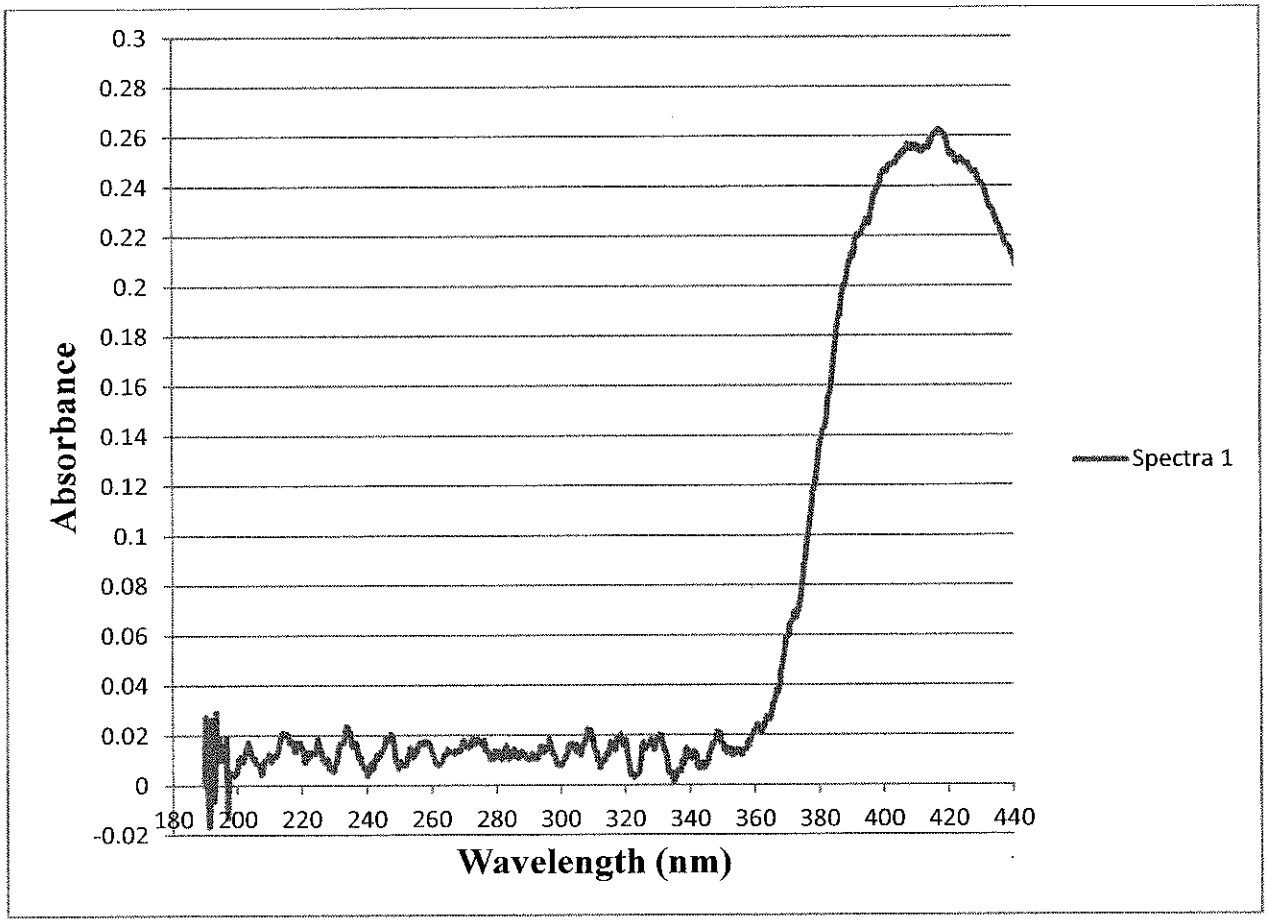


Figure 2: Scatter plot of the single population's major axis' bin to frequency with its Gaussian curve.

My comments: When reading a Figure caption it should "stand alone" so the beginning of the caption with the word "Still" implies that I have read something else. Begin with

Frequency plot of the major axis. You will also note that I rearranged the presentation of your graphs to expand the vertical axis on the sheet to highlight it more. I moved two sentences onto the next page. Ideally putting these two graphs on one page allows the reader to cross compare the information in comparing the potatos from 2 (above) to 1 (below) population hypothesis. In order for this to work both plots should have the same "Y" axis value (that is 0-8) and be given the same amount of page space. This way the reader will be able to mentally superimpose the plots when reading.

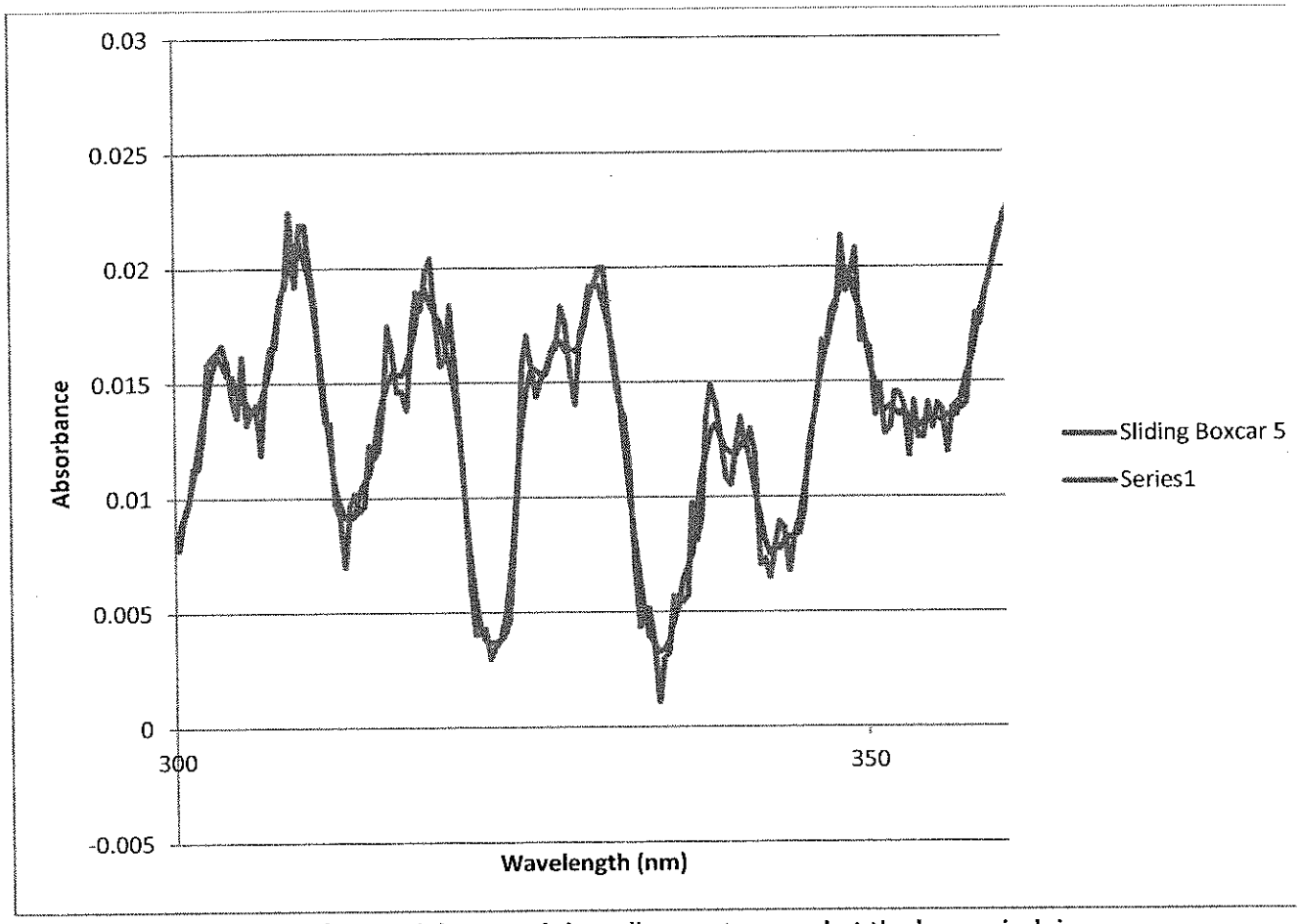
EXAMPLE 2



My comment was: Nice graph. The number is correct, the spatial use of the page is good because it allows the eye to "see" well. The size of fonts is appropriate and the units are present. You have also "blown" up the graph to make the "noise" apparent, which is relevant because it is the actual focus of the filters. Good job.

What did I miss?

EXAMPLE 3 (what did I miss?)



My comment was: I like this graph because it is really easy to see what the boxcar is doing.....

☺

CRITIQUE THIS GRAPH

machine. The first spectrum constructed was of the first method blank, including the cuvette containing unreacted dithizone and buffer, Sample 1. This spectrum was then plotted against fitted Gaussians (deconvoluted data) and the sum of the Gaussians as seen in Figure 1. The sum is plotted in order to determine how well the deconvoluted data fits the raw data.

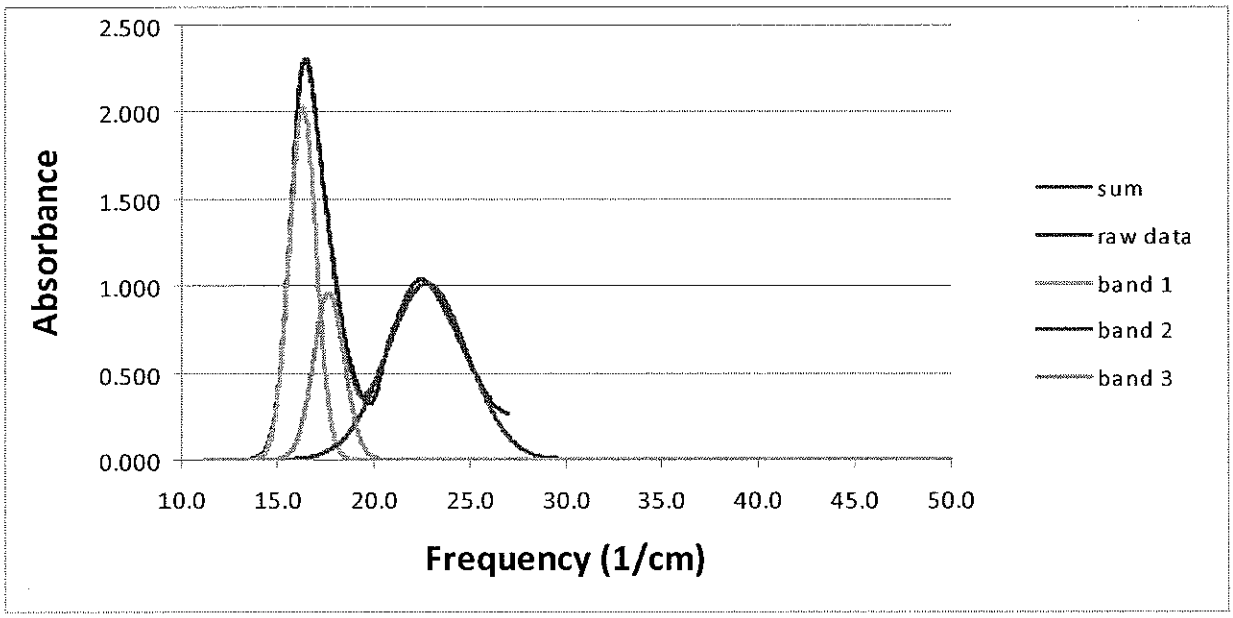


Figure 1

POSTER (LAB FINAL)

Local Lead Results: Your presentation for your local lead project must clearly define the research question, present your individual results with an evaluation of the quality of your presented data:

- What instrument was chosen? What is the expected MLD for that instrument and associated preparation steps? Does the method limit of detection allow you to determine if a sample is above or below any appropriate regulatory standards?
- How was the instrument calibrated?
- What is the quality of the calibration?
- What are the calculated concentration of your fortified field blank and laboratory duplicates? Include the confidence limits obtained from the quality of the calibration used?
- Using those values what is the % recovery for your fortified sample? Does it fall within the acceptable range? Does the relative difference for the duplicates meet acceptable standards when considering your method limit of detection?
- Compare your quality of results to those obtained by other students working on other samples. Are your values acceptable or should any of your (or other students) data "outliers"? What tests did you use to come to that conclusion?
- For the collected collaborative data, excluding outliers, create a table, graph, or map that cross compares the values obtained. Reflect on the patterns observed (or anticipated but not observed).
- What questions arose during this process that should be addressed in a second round of research?

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

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

$$LOD_{instrument} << \frac{400x10^{-6}x0.24\,g_{Pb}x5}{50x40\,mL_{analysis}} = \frac{0.24x10^{-6}\,g_{Pb}}{1\,ml_{analysis}} = 0.24\,ppm$$

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.

The data must be displayed in a map form in the context of all other data for the site. The relative significance of the data to the full site data must be discussed. That is sampling and its significance at the site and within the lab must be discussed.

HOW TO WRITE A SCIENTIFIC POSTER

http://www.ncbi.nlm.nih.gov/pmc/articles/PMC1955747/

http://web.grinnell.edu/individuals/kuipers/stat2labs/write%20paper.html

http://www.stanford.edu/group/blocklab/dos%20and%20donts%20of%20poster%20presentation.pdf

Here is an **excellent site** that allows you to download poster formats as ppt templates. Select a format that appeals to you and is consistent with the printer capacities available to you.

http://www.makesigns.com/SciPosters_Templates.aspx

Loyola's IC has poster printing capacity as described at:

http://www.luc.edu/digitalmedia/anytimeanywhere/posterprinting.shtml

INSTRUMENTAL STUDENT RESEARCH JUDGING SHEET
Adapted from the 2013 Clay Minerals Society Meeting

Directions: Please circle the most appropriate scale value for each question. Calibrate your scores relative to previous poster presentations. Provide comments when applicable.

		Poor	Fair	Ave	Good	Ex.
1	Was the presentation professional?	1	2	3	4	5
	Was the poster readable?	1	2	3	4	5
	How involved was the student with this project?	1	2	3	4	5
	Was the poster well organized?	1	2	3	4	5
	Did the student provide well-informed responses to the questions?	1	2	3	4	5
	How knowledgeable was the student about this project?	1	2	3	4	5
2	Was the introduction/literature review sufficient and relevant?	1	2	3	4	5
3	Was the study well designed?	1	2	3	4	5
	Was the purpose clearly stated	1	2	3	4	5
	Were the statistical procedures appropriate?	1	2	3	4	5
	Were the conclusions valid based on the results of the study?	1	2	3	4	5
	Rate the scientific impact of the research.	1	2	3	4	5
4	How well did the student prove quality control?	1	2	3	4	5
	Did the student address MLD for the instrument	1	2	3	4	5
	Was instrumentation calibration well described and accurate?	1	2	3	4	5
	Was a % recovery from the fortified blank included and how valid was it?	1	2	3	4	5
	Did the student compare their data to the aggregate data and comment on its validity?	1	2	3	4	5
	Did students use an appropriate statistical test to validate their data quality with respect to the class data?	1	2	3	4	5
	Was a site map with results provided?	1	2	3	4	5
	Were future research questions addressed in an adequate fashion?	1	2	3	4	5