INSTRUMENTAL ANALYSIS LABORATORY MANUAL

Chemistry 315
Fall 2009

TABLE OF CONTENTS

INSTRUMENTAL ANALYSIS LABORATORY POLICIES

CHEMISTRY 315 – FALL 2009

INTRODUCTION

In the laboratory component of Instrumental Analysis you will be exposed to several different types of instrumental techniques. In contrast to CHE 215, where the emphasis was on quantitative analysis, this course focuses on the principles of operation and the strengths and limitations of different types of instruments. In most experiments, you will be investigating the performance characteristics of the instrument in addition to using it to make a measurement. It is still important, however, that you continue to use your best analytical technique when preparing standards and samples. **Poor technique will always produce poor results!**

LABORATORY MODULES

The laboratory course is divided into five modules, each of which emphasizes a different aspect of instrumental analysis. Each module consists of 1-3 experiments that relate to the theme of the module. You will work as part of a 3-person group, but because of limited instrument availability there is a possibility that you will have to schedule analysis time outside of class.

LAB PREPARATION

The key to efficiency in the lab is preparation. You are expected to read the lab and contact the instructor or the TA **before** the lab period for questions and/or clarifications. We highly recommend that you consult with your group members before the lab period to divide responsibilities so that your laboratory time is used most efficiently. It is also important that you are considerate of your lab partners and arrive at lab on time.

LAB NOTEBOOK

A hard-bound, carbonless scientific lab notebook (National Brand 43-644 or similar) is required for the lab. The notebook is available at the Alamo, Barnes & Noble, and at several online sites.

You must keep a lab notebook starting with the first day of experiments (January 20, 21, or 26). **All data must be recorded in the laboratory notebook, not on loose sheets of paper or on the lab manual.** At the end of each laboratory period, you must sign each page and then turn in the duplicate page(s) to the TA. The lab notebook will be graded each week and be worth 10 Points, or about 16% of your lab score. **This page(s) is due when you leave the lab; you may not leave, fill in or transcribe data and then turn in the page(s) later. Late lab pages are worth 0 points.**

The primary purpose of the laboratory notebook is to record data and experimental details, especially those that are not included in the lab manual or are deviations from the lab manual. It is good practice to write detailed procedures, but it is not required. The notebook will be graded based on the following guidelines:

1. The notebook has the few pages reserved for a table of contents. This table of contents must be kept up to date.
2. Fill in all of the information boxes on each page.
3. No pages (other than the duplicate pages) should be removed for any reason.

4. All data entries must be in indelible (i.e. non-erasable) ink.
5. Mistakes and errors should be crossed out with a single line. White-out is not allowed.
6. Try to keep the notebook reasonably neat – you should be able to understand each entry. It is often helpful to construct tables prior to lab and fill them out as you collect data.
7. Keep your notebook current. It is unethical (not to mention illegal in many circumstances) to go back and fill in your notebook with data you wrote on loose pieces of paper. Backdating (i.e. going back and writing the date you think the data was recorded) is also forbidden.
8. Each week (excluding lab lecture weeks) your notebook pages will turned in and graded for a total of 10 points. You will be graded on the following:
    - Neatness: 3 Points
    - Completeness: 5 Points
    - **Each page** dated and signed: 2 Points

    You must not write directly on the duplicate pages; they should be an exact copy of your lab notebook.

BONUS POINTS OPPORTUNITY

You can earn up to 20 extra credit points by adopting good laboratory habits. You will be given a score of 0 to 10 points in two categories: safety and preparation. **Since these are bonus points, they will be awarded very sparingly. You are not entitled to these points; they are rewards for excellent safety consciousness and preparation.** Following are some guidelines for awarding these points:

- **Safety.** You must consistently observe laboratory safety policies. The most common and serious violation of safety policy is not wearing safety glasses. **You must wear your safety glasses at all times without reminders**, even when you are just sitting at an instrument waiting for data. If you must be reminded to put on your safety glasses, if you listen to an MP3 player (or other audio device), or you play games or text message with your phone during lab, you will automatically be given a safety score of 0.
- **Preparedness.** You must arrive at lab on time and be ready to begin work immediately. You should have read the lab **before** class. Being tardy, working on a previous week's lab report during class, not reading the lab manual, printing the lab manual during lab, or having an unexcused absence will automatically result in a preparedness score of 0. Good preparation will pay dividends well beyond these bonus points; you will finish your lab work much more quickly and you will likely achieve better scores on your lab reports.

DATA ANALYSIS: SPREADSHEETS

You will be expected to use a spreadsheet to work up your data. It is wisest to use Microsoft Excel, as it is available at all campus labs and all of the textbook examples use Excel spreadsheets. ***If you are not yet comfortable with Excel, it is well worth your time to learn how to use it.*** You might save hours of calculator time on some lab reports if you are proficient with Excel. Work through the online tutorial on the course web page (www.che.ilstu.edu/jebaur/315/handouts/excel_training.pdf) or teach yourself through the examples on the textbook's web site (see course home page for the link). For each lab report, you will be required to turn in a table of all data and calculation results. If you use Excel, you can simply email your spreadsheet(s) to the TA by the due date.

Important note regarding group spreadsheets and academic honesty. *While it is permissible for a group to tabulate data together, the calculations and final results should be individual efforts. Do not simply turn in the same spreadsheet for each group member, or you will receive a 0 for that portion of the lab.*

REPORTS

You must write a report for each week of lab. These reports are due one week after completing the lab. These are not full formal reports, but rather are concise summaries of the lab including tabulated data, results, calculations, and answers to the questions. These reports are worth 50 points (except for Module III, which is worth 70 points). The late penalty is 10% per school day (maximum 50% deduction).

IMPORTANT

- Reports are due at the beginning of the lab period. **YOU MAY NOT WORK ON YOUR LAB REPORTS DURING THE LAB PERIOD.**
- You are encouraged to work with your partners on analyzing your data. However all written work must be individual efforts. Your report must be written in your own words and contain your own calculations and interpretations. Copied reports will be penalized severely.
- Lab reports will not be accepted from anyone that missed an experiment. You may not simply copy your partners' data and turn a report; you must arrange to do the experiment (preferably during another lab period).

GENERAL REPORT FORMATTING

Reports must be printed with a high quality printer and have a title page containing the information shown on page ix. One member of your group must attach your copies of the raw data.

- Make a cover sheet for every report. See the example on page ix.
- **On every page of the report** include a header or footer with the page number and your name.
- Tabulate all data and results of calculations. If done in Excel, these tables and calculations can be turned in by email (send to your TA), **but all files must be turned in by the due date for the written portion**.
- Assemble your report in the same order as the Report Checklist for the experiment. **If any of the graphs or tables listed in the Report Checklist are included as appendices, you must provide a reference to them in the report.**
- Answer all questions completely and in paragraph form. Do not merely answer "yes" or "no", but always give a rationale for your answer. Here is an example of an answer that would receive full credit:

  What is the optimal flame observation height for Zr?

  The data in Figure 3, a plot of Zr absorbance as a function of observation height in the flame, shows that the maximum absorbance occurs at a height of 2.25 cm. Therefore, the best sensitivity for atomic absorption of Zr occurs at an observation height of 2.25 cm.
- Be sure to include sample calculations. Handwritten calculations are acceptable. Note: if you use Excel for your calculations, we still require written sample calculations.
- Sample calculations should be easy to follow. For example:

  Sample calculation of Zr^{4+} stock solution concentration:

$$\text{Zr (ppm)} = \left(\frac{2.3044\text{ g } ZrCl_4}{1\text{L}}\right)\left(\frac{\text{mole } ZrCl_4}{233.03\text{ g } ZrCl_4}\right)\left(\frac{1\text{ mole Zr}}{1\text{ mole } ZrCl_4}\right)\left(\frac{91.22\text{ g Zr}}{1\text{ mole Zr}}\right)\left(\frac{1000\text{ mg}}{1\text{ g}}\right) = 902.1\text{ mg/L} = 902.1\text{ ppm}$$

- When fitting data to a line (e.g. a calibration curve), always use linear regression. Report the equation for the line and the correlation coefficient (r or r^2).
- For all replicate determinations, the average, standard deviation, and % relative standard deviation must be reported.
- Watch significant figures!
- Always calculate a % error when comparing results to a literature or known value.
- Following are examples of acceptable presentation of data. Note that tables include descriptive captions and that columns include units.

Table I. Data for calibration curve of Zr by atomic absorption spectroscopy

Standard	Concentration (ppm)	Absorbance (AU)	RSD (%)
1	1.02	0.020	3.2
2	2.04	0.041	1.8
3	5.10	0.110	5.1

Equation of the Zr calibration curve: Absorbance = 0.022[Zr] - 0.0031 (r^2=0.9987)

Mass of $ZrCl_4$ for standard preparation: 2.3044 g

Table II. Results of analysis of Zr in alloys by atomic absorption spectroscopy (Results are averages of 5 replicates)

Sample	Average Concentration (mg/kg)	Standard Deviation (mg/kg)	RSD (%)	CL (95%)
A	3.62	0.29	8.0	3.62 ± 0.36
B	7.93	1.15	14.5	7.93 ± 1.43
C	8.01	0.89	11.	8.01 ± 1.1

Following is an example of a poorly-constructed table. This table does not have a descriptive title, lacks units, has poor labeling of the samples, has totally ignored significant figures, is poorly aligned, and has no column for %RSD.

Table II. Data for part 3b.

	Conc.	Std. dev
Joe	3.6232093	0.29325333
Bob	7.9332301	1.15352342
Katie	8.01328928	0.89834569

GRAPHING

Good graphical presentation of data is critical for analysis of experimental results. For the sake of clarity, conciseness, and consistency, it is required that you follow proper graphing convention. Listed below are general guidelines for properly plotting and annotating a graph.

1. Choose a scale that best shows the full dimensions of the data and also results in scale divisions that allow easy interpretation of the data. In other words, do not bunch the data in one corner of the graph. Most spreadsheets scale graphs automatically, but you can usually re-scale the graph manually. The origin need not be included in the graph unless you are showing data near the origin.
2. Plot the dependent variable (the one that is a function of the other, such as absorbance as a function of concentration) on the vertical axis. The independent variable should therefore be plotted on the horizontal axis. **Always label both axes and include units in parentheses**.
3. Each point should be located with a small distinct data point. Use different symbols (e.g. circles, squares, etc.) to distinguish one data set from another.
4. Lines through data sets should not "connect the dots". The best method (if the data is supposed to be linear) is to use linear regression to determine the best-fit line for your data.
5. **Never let Excel draw a smooth line through your data.** In nearly every case, these curves are meaningless!
6. When you plot data over several orders of magnitude, it is advisable to construct a log-log plot so that data at both low and high concentrations are visible. Although you can do this by setting the properties of the axis in Excel to "logarithmic", it is usually best to calculate the logarithm of the data before graphing (especially when you want to make a linear fit to the log-log data).

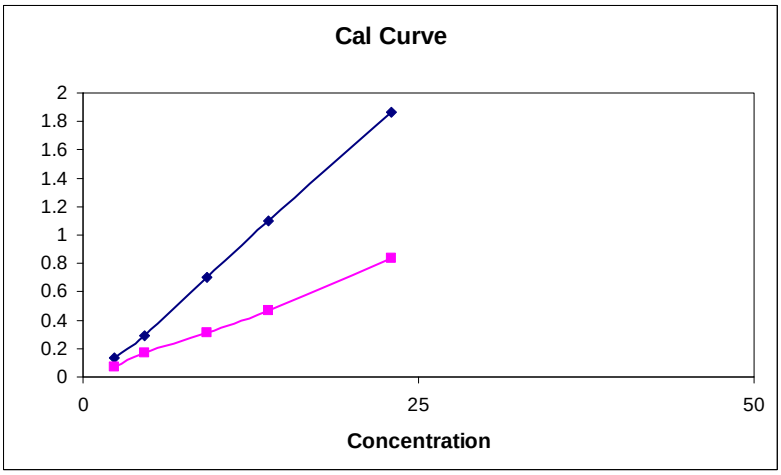

Figure 1. Example of a poorly-made graph. Note poor title, lack of *y*-axis label, lack of units on *x*-axis, missing legend, poor choice of *x* scaling, and smoothed lines connecting the data points.

RULES FOR GRAPHING IN INSTRUMENTAL ANALYSIS

1. Graphs may be inserted in the text of the lab report. However, be sure the graph is large enough so that the data is easily interpreted, and that axis labels are legible. If you include the graph as an appendix, include a reference to it in the main body of the report.

2. Every graph should be numbered and have a have a caption that completely describes the graph.
3. Multiple sets of data may be plotted on the same graph as long as the axes are appropriate for all sets of data. A legend must be included when multiple sets of data are plotted.
4. Graphs must have titles, axis labels, and units.
5. The equation of any regression lines should be included on the chart.

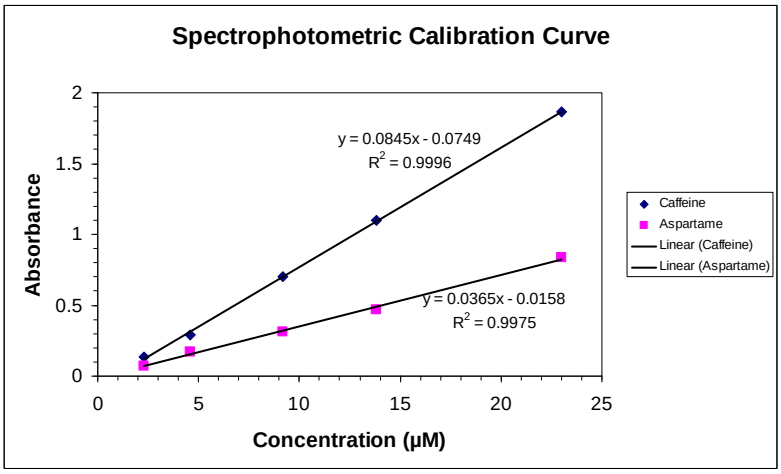

Figure 2. A correctly plotted graph.

An example of a poorly-constructed graph is shown in Figure 1. Figure 2 shows an acceptable graph.

GRADING CHECKLIST FOR GRAPHS

Graphs that you turn in as part of your report will be graded according to the following checklist. Up to 5 points can be deducted for failing to meet these guidelines.

- ☐ Graph includes descriptive title
- ☐ Axes are labeled with units in parenthesis
- ☐ Legend is included (if more than one set of data is plotted)
- ☐ Data points, labels, and legends are large enough to be legible
- ☐ Regression data (equation of line and R^2) included for linear plots
- ☐ Smooth lines are not used to connect data points

ADDITIONAL INFORMATION

- You are expected to attend every laboratory period. Notify the instructor and your group members in advance if you cannot attend lab.
- You should be in lab and ready to start at the beginning of the lab period.
- Your work area, the instruments you use, and community areas (e.g. balances, chemical storage, etc.) should be left clean at the end of each lab period.
- Strict adherence to safety guidelines is expected at all times. **Always** wear safety glasses when in the lab and know the locations of the eyewash, the safety shower, and the fire extinguisher.
- **Listening to iPods, MP3 players, etc. is not allowed in the lab. It is a safety risk and a serious impediment to group communication.**
- **Cell phones should not be used in the laboratory.** If you must answer or make a call, go to the hallway. Keep your calls as brief as possible. **Also, please no texting in the lab.**

- **You must use your best analytical technique!** Poor results are almost always a result of poor technique. Instruments can provide data that is only as good as the samples provided to them. In the words of the ancient instrumental analysis proverb: **Garbage in → garbage out!**
- Reagents necessary for your experiment will be provided in a common area. Since as many as 3 different experiments may be in progress on a given day, and we share the laboratory with CHE 215, it is vital that you double-check the label before taking any reagent. For the same reason, be completely sure that you are putting waste in the appropriate waste container.
- Always calculate the amount of reagent you will need before taking it from the bottle. We have determined how much should be required for everyone to do the experiment and take a small amount extra. If you take much more than required, we will run out of reagents. In addition, this wastefulness presents a disposal problem.
- Never contaminate provided solutions by inserting pipettes into the reagents. Pour what you need into a clean beaker, and then pipette the required volume. Dispense the unused reagent into the appropriate waste bottle or flush down the drain with plenty of water (with the instructor's approval).
- Read the required material and do any necessary calculations before coming to lab. In other words, PLAN AHEAD!

(Sample Cover Sheet Required for All Reports)

MODULE VII

ELECTRON SPECTROSCOPY

AUGER ELECTRON SPECTROSCOPY
EXPERIMENT VIIA

Gary Gnu

Chemistry 315

Experiment Performed
November 31, 2009

Group 7E
Lab Partners: Christopher Glenn, Fran Allison

UNKNOWN #Y13