

# Analytical Chemistry Lab Reports

Format and Calculations

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# Measurement

- Analytical chemistry is entirely about measurement, what these measurements signify, and the understanding concerning the quality of these measurements.
- In this class, you will be performing a number of experiments to measure the concentrations of various analytes and performing statistical analysis on your data.

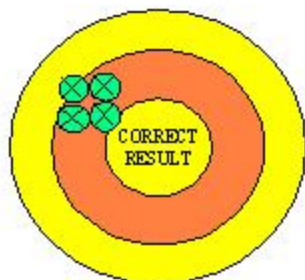
# Quality of Measurements

- In each experiment, you will obtain sets of data.
- This data will be statistically analyzed to measure the quality of the data.
- Accuracy and Precision are the two broad modes utilized in determining the quality of your measurements.
- Statistical Analysis allows you to mathematically determine a higher relative degree of accuracy and precision contained within your data.

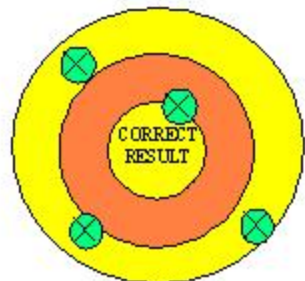
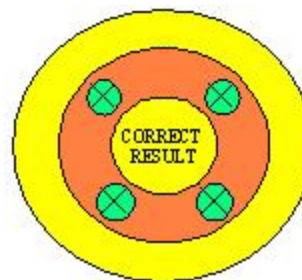
# Accuracy and Precision

- Accuracy is how close the mean (average) of all your data points is to the actual (known) value. Reflects the degree of systematic error in your experiment.
- Precision is a measure of the variability of all the data points—the degree of consistency in reproducing the same data points. Reflects the degree of random error in your experiment.

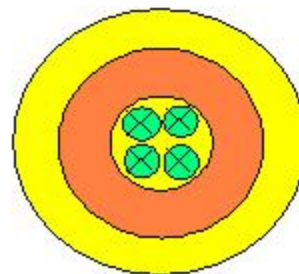
PRECISE BUT INACCURATE



ACCURATE BUT IMPRECISE



INACCURATE AND IMPRECISE



ACCURATE AND PRECISE

# Statistical Analysis

- It should become intuitive when to use what types of statistical analysis as the semester continues.
- Understanding the concepts behind what each applied statistic model reveals is the key to good statistical analysis—and for that matter, this is one fundamental key to sound scientific ability.

# Report Format

- Title of Report
  - Author(s)
  - Section
- Abstract
- Introduction
- Experimental
- Results
- Discussion
- Reference
- Appendix

# Abstract

- What has been performed in the lab.
- Summary of results and conclusions.
- Report what you did and what you observed.
- Brief, brief, brief!!!!
- 2 sentences. 3 maximum!!!!!!!!!!!!

# Good Abstract

“A single solution of unknown  $\text{Cl}^-$  from  $\text{NaCl}$  in water was aliquoted and titrated with  $\text{AgNO}_3$  through Fajans method. It can be said with 95% confidence that the true mean of the percent weight of  $\text{Cl}^-$  from the titrated samples was  $59.6309\% \pm 0.2479\%$ .”

- Very specific.
- What was done. What was discovered.
- Very brief. 2 sentences.

# Bad Abstract

“The main objective of this lab was to determine an unknown chloride ion concentration by the process of titration with a known concentration of silver nitrate solution. The lab performed the standardization using Fajans method with adsorption indicator that was dichlorofluorescein as indicator. “

- Never start an abstract by describing the “objective” of the experiment.
- Say what you did, and what you found.
- This does not specify what was observed or discovered.
- The indicator is a detail which belongs in the experimental section.



# Introduction

- Address the experiment and/or the problem being investigated.
- Address the significance of the task at hand.
- Discuss the concepts/theory involved in the experiment.
- If you choose to discuss the history of the chemistry, or applications, this is where you do it.

# Good Introduction

“A Beer’s Law Plot and a standard addition plot are analyzed in this experiment. These two methods are used to determine the concentration of an unknown  $\text{Fe}^{3+}$  solution. The latter plot unlike the former one considers the matrix effect—whereby the wavelengths of light emitted from other ions besides the standard, interfere with the absorption of light from the  $\text{Fe}^{3+}$  standard (or another ion of interest). We must therefore account for the matrix effect if the two concentrations of the unknown are statistically different. We can determine whether there is statistical difference between the two results using both the t-test and the f-test. According to Beer’s Law, the absorption of the species of interest is directly proportional to the concentration of the species. The species of interest for this experiment are both the standard  $\text{Fe}^{3+}$  and the unknown  $\text{Fe}^{3+}$ . Beer’s Law accounts only for monochromatic radiation, so care must be taken so that the solutions used for each plot are dilute.”

- The objective of the lab is pointed out, the theory behind the experiment and why it works is clearly, succinctly explained.

# Bad Introduction

“Spectrophotometry is used to determine the concentration of a solution of  $\text{Fe}^{3+}$ . An iron-containing tablet is crushed and mixed with concentrated HCl. Water is added, and then filter paper is used to transfer the iron solution to a 500ml volumetric flask and diluted with distilled water.  $\text{Fe}^{3+}$  is very weakly colored. 2,2'-dipyridine is added to the solution and forms an intensely colored complex with  $\text{Fe}^{3+}$ . Standards of varying concentration are prepared and the absorbances measured. “

- The far majority of this Intro belongs in the experimental section. Do not list your procedure in the introduction.
- This intro doesn’t include the theory of Beers law nor how it is used or what it can tell us—which is the main concept/theory behind the experiment.

# Good Experimental Sample

“**6.05 mL** of 50% NaOH solution was added to the shoulder level of a 1-L bottle using a 10-mL graduated cylinder. The solution was mixed inside the closed container.

**25.00-mL** of white vinegar was dispensed into a 250-mL volumetric flask using a pipette, and distilled water was added to the mark. **25.00-mL** aliquots were dispensed to each of the five 250-mL Erlenmeyer flasks using a **volumetric pipette**.

**50.04-mL** of distilled water and 3 drops of phenolphthalein indicator were added to each flask.....

- Experimental parameters were recorded according to what you, the experimenter, did.
- You must record what you specifically did, especially the deviations from the experiment instructions (e.g. The actual volumes and weights you took).

# Bad Experimental Sample

“The experiment was started by preparing our NaOH solution with **6 mL** of 50% NaOH solution and diluting to the mark. It was used throughout the experiment to fill the burette for titration. Five Erlenmeyer flasks filled with diluted vinegar and five flasks with weighed portions of KHP were titrated with the NaOH solution. “

- This experiment section does not display the actual volumes and amounts that you used in the experiment.
- This looks like the student just simply copied the lab procedure. Do Not do this!
- The experimental section displays precisely what YOU did, not what the procedure tells you to do.

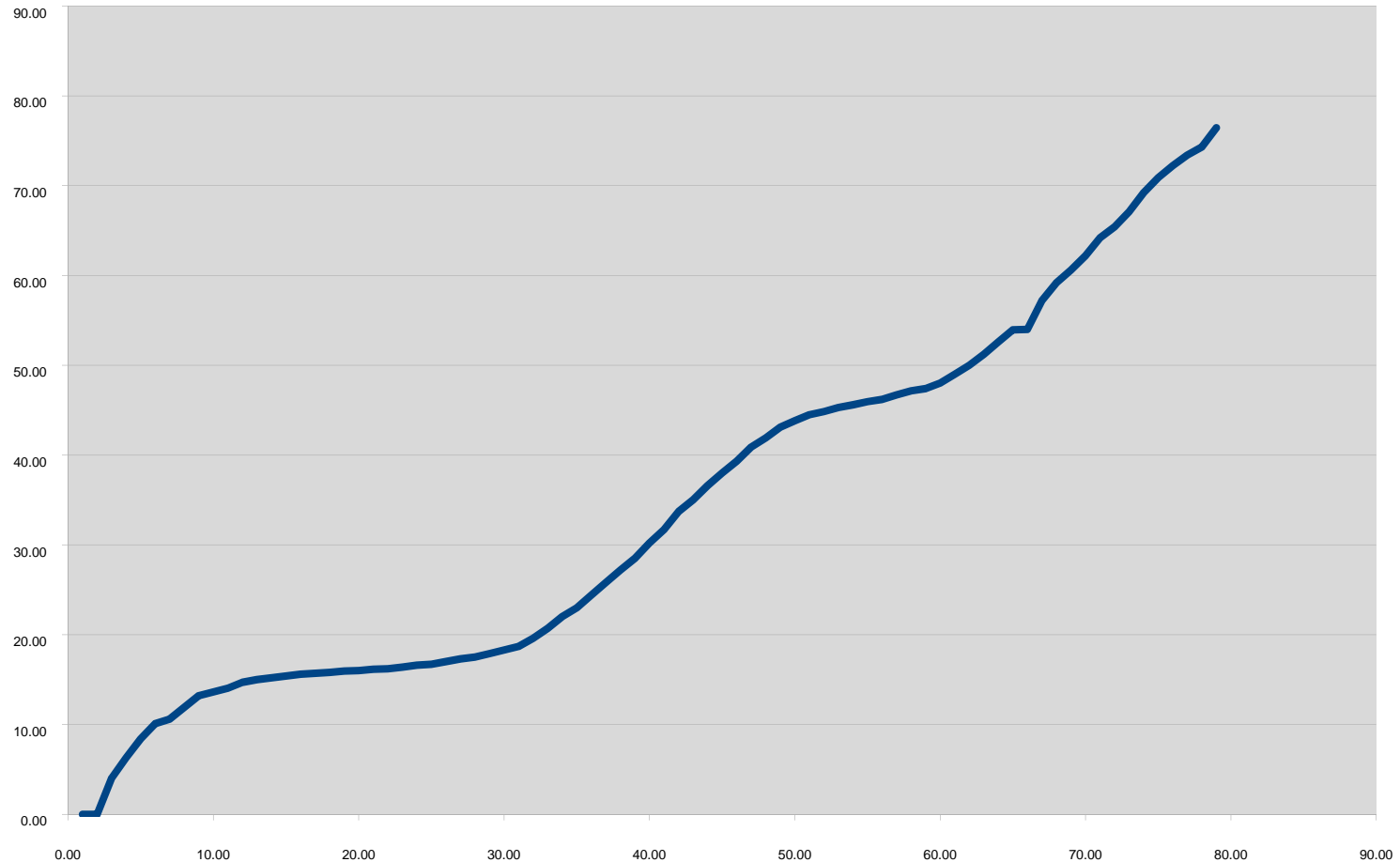
# Good Results Section

- Include finalized and tabulated results that are not already in the excel spreadsheet.
- Cite the excel file for all other data.
- Include finalized graphs/charts (with proper formatting, titles, labels, etc.) your formal report.
- Make sure graphs are large enough so we can clearly see the relationship between the data points!
- Include any detailed data calculation, error analysis, etc. that you did not already perform/show in the excel file.
- Perform long calculations in the appendix , yet cite them in the results section.

# Bad Results Section

- DO NOT COPY AND PASTE THE SAME INFORMATION FROM YOUR EXCEL SPREADSHEET INTO YOUR FORMAL WRITTEN REPORT!
- Do not forget to cite all other data/results that have been accrued in your excel file.
- DO NOT LIST AND ANSWER THE QUESTIONS ASKED IN THE EXPERIMENT. SEE DISCUSSION SECTION FOR THIS.
- If we gave you a template sheet or template area for data or graphs in the excel file, USE IT! Otherwise, create it yourself in excel or import the properly formatted data/graphs into your formal report results section.

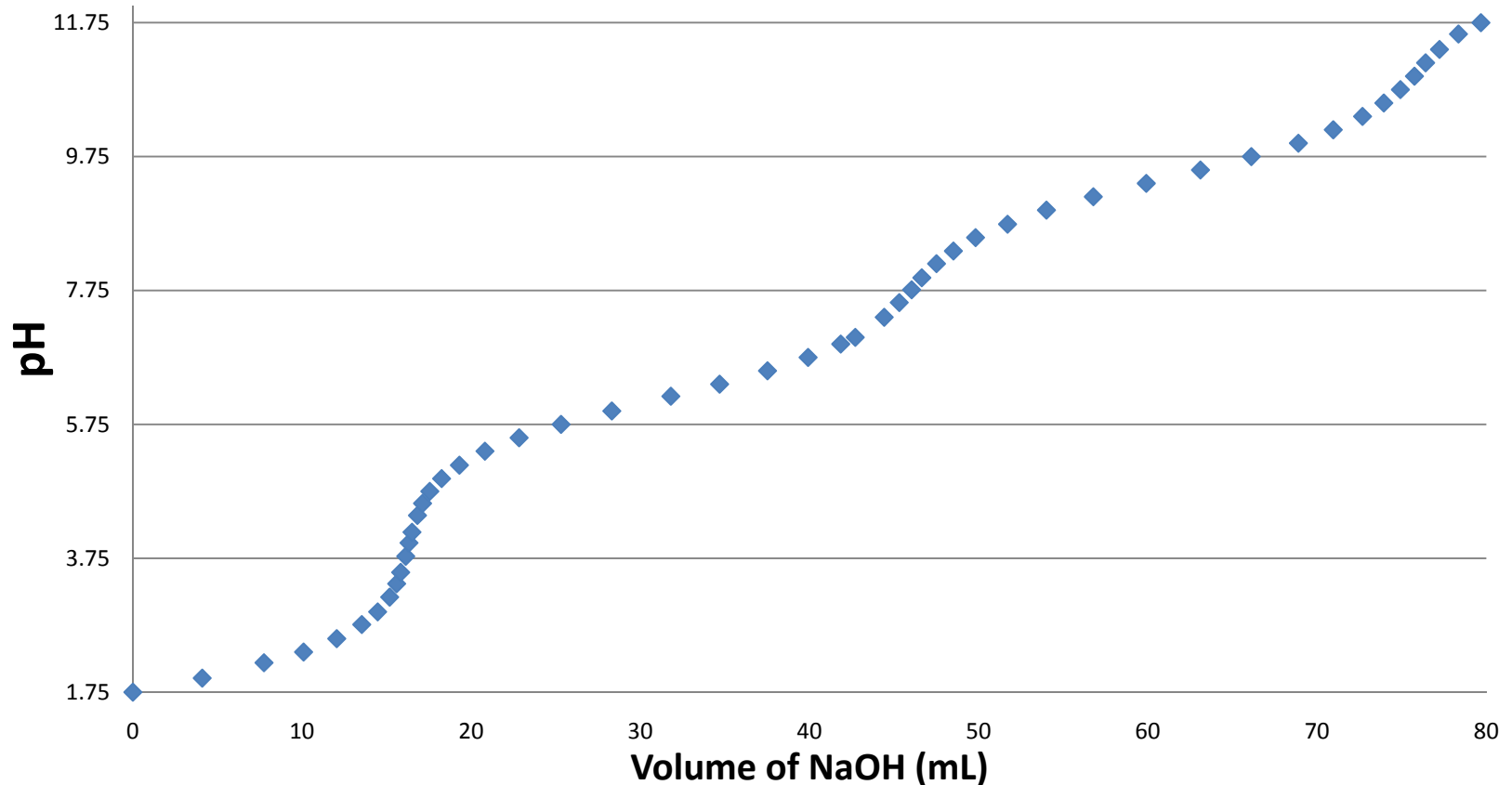
# Inadequate Graph



- This graph has no title, no axis labels, no units, the numbers are so small I can't read them, no descriptive factors.
- Always use data points instead of lines. Only use lines when fitting a regression line to your data.
- I have no idea what I am looking at.
- This graph would not get a good score.

# Adequate Graph

pH curve of NaOH-Histidine Titration



- Graph is clearly labeled with a scale that allows me to identify the continuity of the data points as well as individual x/y points.
- No outliers on the graphs.
- The main data points go through the center of the graph.

# Good Results Sample

## Data/Results

My group data: the chloride sample mass = 1.4704 g = 1470.4 mg

Flask #	Initial reading (mL)	Final reading (mL)	Color	Volume (mL)
1	1.50	34.33	Pink	32.83
2	1.50	34.26	Pink	32.76
3	1.30	34.20	Pink	32.90
4	0.40	33.18	Pink	32.78
5	0.90	33.75	Pink	32.85

From Cuiyan Zhao's data: the chloride sample mass = 1.1566 g = 1156.6 mg

Flask #	Volume (mL)
1	32.83
2	32.76
3	32.90
4	32.78
5	32.85

See appropriate excel sheets for graphs, calculations, and other tabulated data.

- This person listed only data which was not tabulated already in excel.
- This person also cited where you can find the appropriately formatted and organized data/calculations.
- The template already provides you with the space and proper format.
  - There is no need to duplicate your data .
- Remember to always put units with ALL of your raw and calculated data in BOTH your formal report and the corresponding excel template.

# Discussion/Conclusion

- This is where you get to interpret/discuss your calculated results, and what they mean.
- Do not list / answer the questions in # format!!!
- The excel templates have sections for each of the questions asked in the experiment. You are supposed to solve/calculate/answer the questions in the template—that is what its for.
  - But!!!!!! The calculations/answers to the questions asked in each experiment are key points of your experiment. You should discuss the implications to these questions with regard to your own data and experiment in the discussion section, in report format.



# Bad Discussion Sample

## “Discussion

1. Shows on Excel data sheet1.
2. Shows on Excel data sheet1.
3. For the white vinegar, % acetic acid = 4.551%; for the red vinegar, % acetic acid = 4.918%.
4. Shows on Excel data sheet1.
5. Done by Excel.
6. From the data, I conclude that the percentage of acetic acid in the different brands (white and red) of vinegar is statistically different to the 95 % CL, while % white vinegar = 4.551%;  
% red vinegar = 4.918%.”

- No # format!
- Discuss the implications of your data in light of the questions asked—in paragraph format!
- Discuss areas of possible improvement or different types of error which may have occurred during the experiment.

# Good Discussion Sample

“.....This result **suggests** that my concentration of NaCl, which is 0.00678 M, is very reliable. For three titrations, the solution was very faint in pink, which **suggests that almost an exact quantity of titrant was added to the analyte for titration.** The difference in the amount of both reagents may be a few drops at the utmost. Whereas, my partner’s true mean is  $39.91 \pm 7.57$  for 95% confidence, which means his true mean falls within a much larger range from 32.34 mL to 47.54 mL for the same confidence interval. **Therefore, his data is much less precise than my data.** The standard deviation for my data set is 0.188 mL.....”

- Interpreting and discussion of the implications of your data analysis is the primary point to all of these experiments!

# References/Appendix

- Cite any references used—Your analytical chem book, UMB handouts, Wikipedia, etc. in the references section.
- Use your appendix to type in long calculations and error analysis which you did not or could not perform in excel. Remember to cite these in your results section.

# Excel Instructions

- Use the templates that we give you!!! These make formatting and the timeliness of your reports much to your advantage.
- You should be a master of excel when you are finished with this class. Learn how to use excel! It will help your science career indefinitely!!!
- DO NOT SIMPLY PUT ANSWERS IN THE APPROPRIATE CELLS. I NEED TO SEE THE APPROPRIATE FORMULA TYPED IN THE CELL OR REFERENCED TO THE APPROPRIATE CALCULATION IN YOUR RESULTS SECTION!!
- YOU WILL RECEIVE NO CREDIT FOR ANSWERS WITHOUT THE APPROPRIATE CALCULATIONS.

# Excel Instructions

- This is where the majority of your work and your results will be located.
- Resources:
  1. Step by step excel instructions and basic statistic function explanations:  
<http://alpha.chem.umb.edu/chemistry/ch313/Introduction%20to%20Measurement.pdf>
  2. Text: Quantitative Chemical Analysis, 7th Edition by Harris
  3. Optional: Applications of Microsoft Excel in Analytical Chemistry, by Crouch and Holler.
  4. Your peers
  5. Me
  6. More on Lab Report Format:  
<http://alpha.chem.umb.edu/chemistry/ch313/Report.pdf>
  7. Instructions for 2007 Excel version two variable input:  
<http://cameron.econ.ucdavis.edu/excel/ex53bivariateregressionstatisticalinference.html>
- Excel/Calculation examples.....

# Statistical Tests

## Accuracy

**Mean**  
(Average)

**Confidence Interval**  
(True value lies within  
a given range based on  
SD.)

## Precision

**Standard Deviation**  
(Measures variability  
relative to mean)

**F-Test**  
(Compares Precision  
of two data sets)