CHEMISTRY 31

INORGANIC QUANTITATIVE ANALYSIS

LABORATORY MANUAL

Spring 2017

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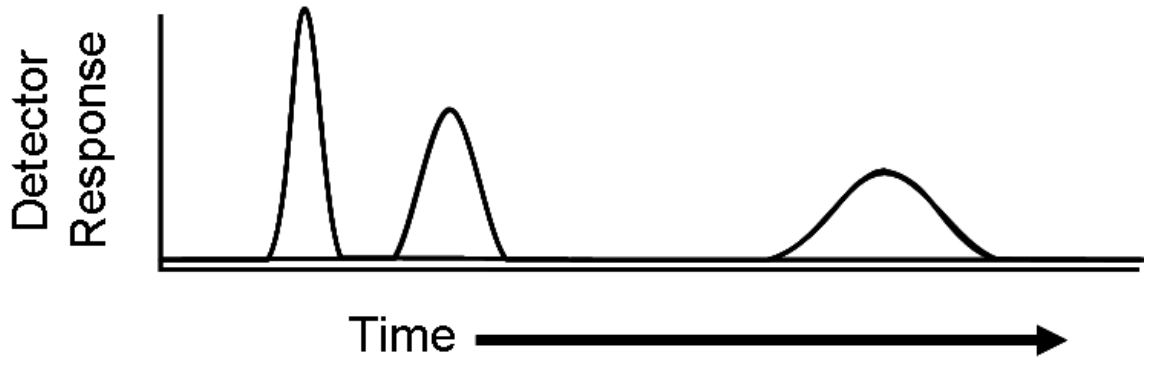

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Acknowledgements: Most of the experiments were developed or improved by past instructors, Dr. Justin Miller-Schulze, Dr. Bradly Baker, Dr. Jahansooz Toofan and Dr. Philip Curb.

COMPUTER USE

Computer expertise covers a wide range of skills, from programming in assembly language to programming in a high level language (e.g. BASIC, FORTRAN or PASCAL) to being a user of application programs. It is the latter, application programming, that is the easiest to learn and yet is extremely useful in this and probably many of your other courses.

Two types of application programs are especially useful: word processors and spreadsheets. It is almost inconceivable that anyone with access to word processing software would prefer to use an antiquated typewriter. Spreadsheets do with numbers what word processors do with words. Since we process numerical data in this course it is essential that we learn to use a spreadsheet.

There are many excellent spreadsheet programs available. While instructions are written for Excel®, other spreadsheet programs work similarly. There are various versions of these spreadsheets, however the most commonly used version is Microsoft Excel®. The exercises below are described with the 2016 version of Excel® in mind. If you are using a different version of Excel®, you may find that some of the instructions given here do not work properly. Consult your lab instructor for additional advice.

1. GETTING STARTED

The following sections give some basic information and instructions as well as some examples you can follow to practice various data operations. Each of the following operations shows what you should type in braces, { }. Follow each typed command with the "ENTER" key. The best way to learn how to use a spreadsheet is simply to try things out. Don't be afraid to select different options even if you aren't sure what will happen. You can use a calculator to check the calculation, and you can always delete what you have done and try again.

2. SPREADSHEET ORGANIZATION

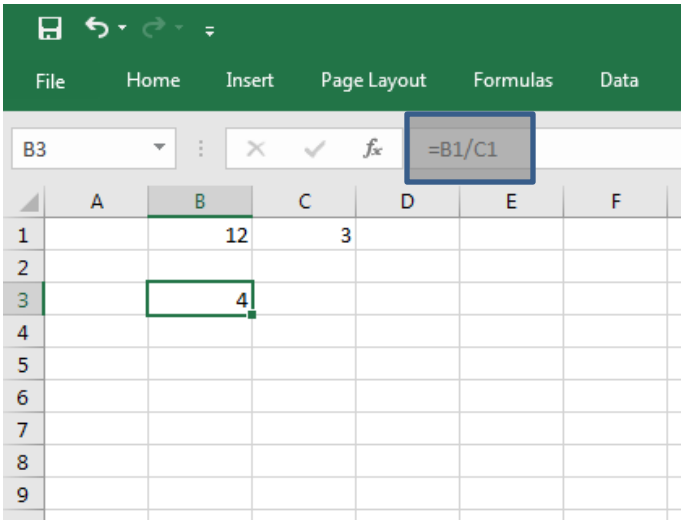
Spreadsheets consist of a number of cells that can be viewed as individual mail boxes, each with a unique address. These cells can contain text, numerical data or computational formulas. The cell addresses consist of a column (letter) and row designation (number). The table below shows typical cell addresses:

A1	A2	A3
B1	B2	B3
C1	C2	C3

It is very important not to confuse the cell address, which is fixed, with the cell contents that are variable. You can move from one cell to another with the arrow keys, the tab key, or the mouse.

3. SPREADSHEET OPERATIONS AND FUNCTIONS

A spreadsheet's primary function is to manipulate numerical data. This is most often accomplished by placing numerical data in certain cells and formulas in other cells. The formulas operate on the data and produce numerical results. Formulas are visible on the command line (see shaded box below) and in the cell while the formula is being entered. Only text, numbers, or the results of a formula are visible in the cell after input has been entered into a cell. The command line always shows what is actually contained in within a cell. For example, in the spreadsheet shown below, the result 4 is returned by dividing the number in cell B1 by the number in cell C1. The result is shown, except in the command line.



Formulas may contain **operators** and/or **functions.** Common operators are those familiar to anyone who has used a calculator: + (add), - (subtract), / (divide), * (multiply), ^ (to the power of), and others. Functions can perform very complex tasks (see below for examples). Formulas may operate on a single cell entry or on a range of cells. Each function must be preceded with the '=' symbol. The colon indicates a "contiguous list" of cells.

Some examples:

=A1+A2 returns the sum of the contents of cells A1 and A2

=SQRT(2) returns the square root of 2

=SQRT(A2) returns the square root of the contents of cell A2

=COUNT(A1:A44) counts the number of entries from cell A1 to cell A44

4. SOME USEFUL FUNCTIONS

A few of the more common functions that we will use in this course are listed in the following table. Remember the contents of the specified address are acted on, NOT the address itself.

Function	Result
=SQRT(X)	square root of X
=SUM(List) () are required	sum of all non-zero list values
=STDEV(List) () are required	sample standard deviation of all non-zero values
=AVERAGE(List) () are required	arithmetic mean of all non-zero values in the list
=DEVSQ(List)	sum of the squares of the differences between an individual values and the mean value

5. BLOCKS

Blocks are groups of cells such as the row A1 to F1, a column A1 to A10, or a rectangular block e.g. the block within the addresses A1 to J10. Many spreadsheet operations require a block designation before implementation. For example, a block of cells must be specified if a **COPY**, **MOVE**, **CUT**, or **DELETE** operation is executed (unless a single cell is involved). Blocks can be selected by "dragging" the mouse over the block.

6. FILE OPERATIONS

If you want to save an Excel® file, click on **FILE** and **Save** and the file will be saved under the current name. If you wish to save the file under a different name and\or to a different drive, use the **SaveAs** command.

Files in Excel®, are designated Book1, Book2, etc. Each Book contains a large number of "pages" designated as Sheet1, Sheet2, etc. Book names can be changed using the SaveAs command in the File menu. Page names can be changed by double-clicking or right-clicking on the page tab.

7. GRAPHICS

Excel® can generate graphs with just a click of the mouse (graphs are called charts in Excel®). To produce a graph, first select the data that you wish to plot. Usually this is organized with x values in one row and corresponding y values in another row to the right. Select the "insert" tab along the top of the Excel® window. Next, select the type of chart

you wish to use to display your data. This will most likely be an x-y or "scatter" plot – this can be selected by finding “Scatter Chart” under “Recommended Chart” or clicking on the icon that shows just axes and data points. Be careful not to select a line chart which looks similar. There are several different formats for the scatter plot available. Usually it is easiest to start with the simplest and change things to your liking later. Whenever an existing chart or object within a chart is selected (highlighted), two additional tabs appear along the top of the window (under "chart tools") for formatting all aspects of your chart. Use the tools provided to make your chart appear the way you want it to appear. Make sure your data appears clearly, the chart and axis are labeled and with units, numbers have the correct number of significant figures, gridlines are appropriate, etc. You may also format specific parts of a chart by right clicking on the part of a chart (dragging the mouse to the appropriate part and clicking on the right mouse button) you wish to format and choosing the format option (usually in the menu box that appears after right-clicking).

Beyond simply formatting the appearance of a chart, additional operations are possible including adding data series, trend lines, error bars, etc. Experiment with manipulating all aspects of your chart until you can confidently make your chart appear as you choose.

Charts can be located either within a sheet of columns and rows, or as their own sheet. Usually, if you wish to print a chart on a single sheet of paper, you should move the chart to its own sheet first. To move your chart to its own sheet, right click on a blank space within your chart and choose 'Move Chart' from the drop down menu. Charts embedded with a sheet can be resized by clicking and dragging the edge of the chart.

8. PRACTICE EXERCISES

As an introduction to spreadsheet calculations you will be given a few simple exercises to perform. In most cases cell contents and formulas will be given. You should duplicate the spreadsheets as shown to gain familiarity with your system. Feel free to experiment by changing data and formulas. These exercises do not have to be turned in, but be sure to consult with the instructor(s) if you have any difficulties. Pay special attention to charts. Charts can be very frustrating to work with, especially when you need to manipulate labels, titles, gridlines, and scales. In some of the lab experiments, you will be required to turn in your results in spreadsheet form. In many cases, a sample spreadsheet covering many of the calculations will be given along with the necessary formulas. You may duplicate the example or modify it as needed. You need to make sure that all of the information requested for the lab report is given (either on the spreadsheet or on laboratory reports).

ALL SPREADSHEETS AND CHARTS ARE TO BE PRINTED ON LASER OR INKJET PRINTERS. NO EXCEPTIONS.

EXERCISE 1

You will begin by creating a spreadsheet to add and to multiply two numbers (12 and 5). The data and formulas are entered into the spreadsheet as shown below. When you type a number, the spreadsheet program treats the entry as a number; when you enter a letter, it is recognized as text. If you wish to enter a number or formula as text, you must change the format of the cell to text. **Set up the example shown below (or**

a similar problem) on a spreadsheet to familiarize yourself with these fundamental operations.

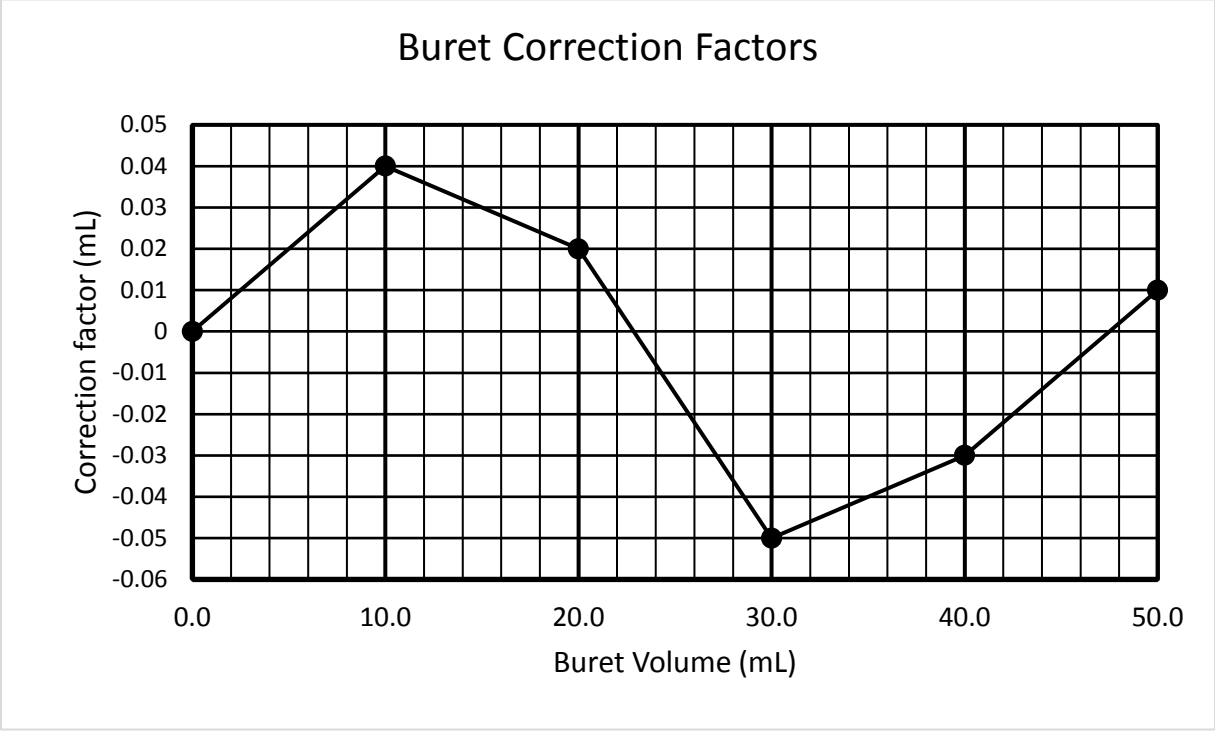
	A	B	C	D
1	12	5	17	60
2	----------			
3	FORMULAS		=A1+B1	=A1*B1

Remember, formulas, data, and text all appear in the "formula bar" as you type. Data, text, and the **results of formulas** appear in the spreadsheet cell after you hit return. Formulas do not appear in the spreadsheet cell! In the above example, the formulas =A1+B1 and =A1*B1 actually occupy cells C1 and D1 respectively. In later examples, formulas and their cell locations will be given.

EXERCISE 2

Experiment with inserting a chart (described previously) to produce a buret calibration curve similar to the example shown below. The spreadsheet used to produce this graph is also shown.

	A	B
3	Volume (mL)	Correction Factor (mL)
4	0	0
5	10	0.04
6	20	0.02
7	30	-0.05
8	40	-0.03
9	50	0.01



EXERCISE 3

A typical application for this course is the calculation of the mean, the standard deviation, and relative standard deviation of a set of data. The results of such an application are shown below. The format for this spreadsheet can be used for the gravimetric chloride experiment report. Set up a spreadsheet similar to the one on the following page. Make sure you get the same answers!

	A	B	C	D	E	F	G
1	Example Spreadsheet for processing of data from Gravimetric Chloride Lab						
2							
3		Gravimetric Chloride Results					
4							
5	Student Name	Lab Section #		Date			
6							
7	Sample	Sample		Precipitate		Mass % Chloride	
8	#	Weight (g)		Weight (g)			
9	1	0.3629		0.7485		51.02	
10	2	0.3714		0.7687		51.20	
11	3	0.3511		0.7255		51.12	
12							
13			Average			51.11	% Chloride
14							
15			Standard Deviation			0.09	% Chloride
16							
17			Relative Std. Deviation			1.74	ppt
18							
19			95% Confidence Interval: +/-			0.22	% Chloride
20		Average value +/- Unc.		**51.1**	**+/-**	**0.2**	**% Chloride**
21							
22	FORMULAS ----------						
23	QUANTITY		CELL LOCATION		FORMULA		
24							
25	Weight % Chloride		F9		=D9*(35.453/143.321)*100/B9		
26	Samples 1 to 3		F10		=D10*(35.453/143.321)*100/B10		
27			F11		=D11*(35.453/143.321)*100/B11		
28	Average		F13		=AVERAGE(F9:F11)		
29	Standard Deviation		F15		=STDEV(F9:F11)		
30	Relative Std. Deviation		F17		=F15*1000/F13		
31	95% Confidence Interval		F19		=F15*4.303/SQRT(3)		
32	Average (correct sig. fig.s)		D20		=F13		
33	95% CI (correct sig. fig.s)		F20		=F19		

Notice that text has been used to identify data and results. It is critical that you use similar headings so any reader can clearly identify the data. You can print out column and row headings and the spreadsheet gridlines by choosing the "Page Layout" tab and then use "print titles" to click the gridlines and headings boxes. To limit the number of digits displayed for the values calculated, you can select a cell or block of cells, right click on the block and choose "format cells". Choose "number" in the left column and then enter the desired number of digits displayed past the decimal point in the "decimal places" box.

EXERCISE 4 MATRIX MATHEMATICS

Matrix mathematics functions are built into most spreadsheet programs. They provide a simple way to solve for the variables (X and Y) in algebraic equations like:

$Z_1 = A_1X + B_1Y$ and $Z_2 = A_2X + B_2Y$

This technique will be useful for the spectroscopic determination of Cr and Co that you will be performing later in the semester. You must construct two matrices from the coefficients in the equations. You "invert" one matrix then "multiply" the inverted matrix times the other matrix. The resultant "product matrix" contains the solutions to the equations. The following directions are for matrix math using Excel.

The example below was taken from pages 464 and 465 of the course textbook.

1. Input the data for the coefficient matrix in a contiguous block.

2. Do the same for the variables matrix.

3. Select a destination block for the inverted coefficient matrix. It must be exactly the same size as the coefficient matrix and not overwrite any needed data.

4. Click on the formula bar and type: =MINVERSE(array).

5. To compute the inversion, depress the ***shift + ctrl + enter*** keys simultaneously. The results will appear in the destination block. This step is sometimes difficult! You must hold down the **shift** and **ctrl** keys before hitting **enter**, otherwise, you will only get one of the inverted values to appear.

6. Select a destination block for the product matrix. It must be the same size as the variables matrix.

7. Use the formula bar or type: =MMULT(array1,array2). The first array is the inverted coefficients matrix and the second is the variables matrix.

8. To compute the product, type together: ***shift + ctrl + enter*** on a PC or command key (⌘) + enter on a Mac. The results will appear in the destination block.

Note! The arrays are ranges: For example the first two arrays shown below are: **A1:B2** and **C1:C2**.

Example

Wavelength	Substance ε $(Mcm)^{-1}$		Absorbance (Z)
	X	Y	
272 nm	**16440**	**3870**	**0.957**
327 nm	**3990**	**6420**	**0.559**

These data correspond to the two equations:

At 272 nm: **0.957** = **16440***b*C_X + **3870***b*C_Y
At 327 nm: **0.559** = **3990***b*C_X + **6420***b*C_Y

To solve for C_X and C_Y:

The "coefficient matrix is:

	A	B
1	16440	3870
2	3990	6420

The "variables matrix is:

	C
1	0.957
2	0.559

The inverted coefficient matrix is:

	A	B
4	7.125E-05	-4.295E-05
5	-4.428E-05	1.825E-04

The resultant "product matrix" contains the solutions to the equations.

	A	B
7	C_X =	4.418E-5 M
8	C_X =	5.962E-5 M

Note that the "b" term drops out. Note also that the product matrix is just B7:B8. Cells A7:A8 are used for identification.

LABORATORY PROCEDURES

I. Introduction

Everything that we have learned about the world around us is the result of observation. It is particularly important that the scientist learns to make his or her observations in a quantitative manner - to attach a number to the property under study. Virtually every aspect of physical reality about which we possess a detailed understanding has been exposed to us through quantitative experimentation.

The purpose of this laboratory is to give you some insight into the principles of making measurements on chemical systems and to permit you to use some of the measurement methods that find frequent application in chemistry. In the process you will learn how to scrutinize a system to find a measurable property that will provide answers to your questions about the system. You will plan some of the experimental procedures and you will use a variety of instruments and techniques. You will analyze your data, draw conclusions and learn to evaluate the reliability of your result. The sum of all these steps should give you a taste of what "doing science" is really like - the discipline of organized thinking and detailed experimentation as well as the excitement of discovering things for yourself.

II. Suggestions for planning your work

At least as much time and effort must be devoted to contemplating the problem and planning the experiment as to the actual execution of the experiment. In the construction trade, this can be summed up as "*measure twice, cut once*". Study thoroughly the description of the experiment before coming to the laboratory. Devise a general plan of attack on the problem. Outline the various steps in the experiment. Think about the details of each step, e.g., sample weight, volume of reagents, measurement of temperature, etc. Make sure that you understand **why** you are carrying out and are not just following directions.

1. Check every experiment to see if there are a series of steps which must be carried out without interruption. If so, careful scheduling is necessary.

2. Some operations are very time consuming; e.g., drying of a sample. Such operations may require little attention, so plan to work simultaneously on other parts of the experiment.

3. Anticipate critical parts of an analysis: Those operations that are prone to introduce serious errors unless carried out very skillfully.

4. It will often be necessary to overlap experiments and, because of limited equipment, work on two experiments simultaneously.

5. Part of your laboratory notebook must be prepared in advance; preparation of a summary data page is a valuable aid in organizing the experimental work. A brief work outline can be prepared if desired, and any necessary

pre-lab calculations need to be carried out before arriving in lab. See the guidelines for laboratory notebook preparation.

6. Be clean and careful. Have your experiment well planned so that you can work with efficiency. Exercise good judgment; don't sacrifice accuracy for speed. Be critical of your work and interpret it honestly. Cooperate with your fellow students and your instructor.

III. Laboratory Notebook

Scientific work requires an authentic record of experimental work and results. The primary record of work you have accomplished in the laboratory is the laboratory notebook. Although neatness and organization are necessary since others familiar with the subject should be able to understand the observations described, immediate and honest written descriptions are the most important factors. The most valuable scientific record is the original, unedited account entered in the laboratory notebook. The following instructions will be useful to you in this course and in all future scientific work:

1. For this course, a bound notebook is required. Original data pages should never be removed.

2. Start the laboratory notebook with a **Table of Contents** that will include the **title**, **date** and **page number** for each experiment. The Table of Contents should be updated as work is recorded. A couple of pages near the front should be used for general reference information such as a safety map of the laboratory, the calibration chart for the buret, constants used in calculations, or your lab partner's phone number. Also, make sure you have your name and lab section number on the notebook. Number all pages of the notebook.

3. Record all observations and data in ink (never use pencil) directly into the laboratory notebook at the time of the experiment. Title each page, including the date of the experiment and when measurements are made. Good recording habits allow one to find mistakes faster or discard improperly collected data.

4. Mistakes should be crossed out, with a single line - NEVER erased or removed with correction fluid (e.g. "White-out"). An error is a part of the experimental record.

5. To help organize the laboratory work and increase efficiency, a "Summary Data Page" should be prepared in your laboratory notebook before beginning each experiment. Some examples are given at the end of the first few sections in this lab manual (see pages 16, 22, 25) to help you, while later on, you will be expected to generate your own tables.

6. Include necessary pre-lab calculations and post-lab calculations in the laboratory notebook.

7. Record qualitative observations. These observations may help you figure out unexpected results later on or to exclude poor data.

8. Data recorded on computers or with scientific instruments such as chromatograms should be taped, glued, or stapled into the lab notebook.

9. Most importantly, while working in the laboratory, you should only record work in your lab notebook. It is unacceptable to record results anywhere else.

10. If the instructor grading the report has any questions about the data in the report, such as when resubmitting a laboratory report, you may be asked to provide the data collected in your laboratory notebook to prove that data is based on actual observations.

IV. Reporting Results: See Appendix I at the end of this manual

V. Care and Use of Chemicals

1. Return chemicals and such shared materials as indicators to the shelves immediately after using them so that your fellow students need not waste time searching for them.

2. IMPORTANT: Spatulas, spoons, pipets, etc. are never to be used to remove any analytical reagent from its container. Measure the approximate amount of a solid or a liquid by pouring the reagent into another container (e.g. beaker or graduate or onto a piece of weighing paper). Never put anything into a reagent bottle. Doing so may contaminate a whole bottle of reagent.

3. Clean up immediately any chemicals spilled when measuring or weighing. Lab benches and the areas around the analytical balances should be scrupulously clean. Cleanliness is an absolute prerequisite for quantitative analysis.

4. Certain reagents, such as solutions for cleaning glassware and concentrated ammonia, need to be kept in the hood. These should only be used in the hood or removed in a beaker covered with a watch glass to avoid spreading hazardous chemicals around the laboratory.

VI. Laboratory Safety

1. Laboratory goggles must be worn at all times.

2. No food or drinks are allowed in the laboratory.

3. You should know the location of safety items in the laboratory. The

instructor will go over these items. Draw a diagram of the lab in your lab notebook showing the location of safety items in the lab.

4. Always wear closed-toe shoes, and avoid wearing contact lenses. Long pants or a long skirt and a lab coat are required to work in the laboratory. You also will be required to purchase and at times use gloves while handling hazardous chemicals (e.g. concentrated acids). Gloves also can reduce contamination of samples (e.g. chloride from skin in the Ion Chromatography Lab).

5. Be aware of what others are doing around you.

6. Fuming or noxious chemicals should not be removed from the hood. Also be careful to avoid dripping these chemicals on the floor or edge of the hood.

7. NEVER pipet by mouth – always use pipet bulbs.

VII. Unknowns

1. The most important component of your laboratory grade is for unknown accuracy.

2. At the beginning of the semester, you will receive all of the unknowns for the laboratory experiments requiring unknowns (most experiments). These will be liquid or powder samples in vials in the unknown rack.

3. All of the unknowns require sample treatment for accurate quantitative analysis. Before you even open an unknown sample vial, be sure that you have received and understand all instructions on sample treatment (from the lab manual, on the unknown vial, and from your lab instructor). Remember that handling the unknown is a rash way (e.g. using a dirty pipet to transfer your water hardness unknown) may result in contamination and a poor accuracy grade. If you believe you have compromised an unknown, you should ask for a replacement. Typically, you are allowed one replacement unknown per semester without a penalty.

4. The two powder samples (for the chloride and soda ash laboratories) need to be dried before use. The unknowns for the water hardness laboratory (which also is used for the AA laboratory), the ion chromatography laboratory, and the spectrophotometric determination laboratory all need to be diluted as instructed on the vials, with the unknown prepared in the volumetric flask being the one for which you will be determining concentrations. The GC laboratory unknown is as you receive it in the vial, but will need to be used with an internal standard added for quantitative analysis.

Balance and Weighing Exercise

We will be using two types of balances in this laboratory. The two kinds of balances, top-loading and analytical, differ in terms of their precision and capacity. The analytical balances have a listed best precision of ± 0.0001 g or ± 0.1 mg and a capacity of 200 g. Note that the precision decreases as one weighs heavier objects. The top-loading balances have a capacity of 1200 g and a listed precision of ± 0.01 g. The top-loading balances are used for weighing out bulk samples of solid and liquid reagents and for other measurements where the higher precision of the analytical balance in not required. A microprocessor is incorporated in these balances. It controls the balance and minimizes the steps required by the operator during the weighing operation. These are "digital" balances. They show the mass on a lighted digital display in decimal form. The instructor will explain the principle of operation of these balances and demonstrate their proper use.

These balances are rugged and easy to use, but can be damaged if abused. Each student will be assigned to a specific analytical balance for the duration of the semester to minimize effects from any inaccuracies. In most cases, two students in each lab section will share a balance. It is the students' responsibility to keep the balance and its surrounding area clean at all times. Report any malfunctions to the lab instructor.

I. Balance Rules: The following are some "DO'S and DO NOTS" that apply to the balances.

DO NOT:

1. Weigh chemicals directly on the pan. (Use an appropriate container: a beaker, a weighing bottle, or a weigh boat).

2. Weigh hot (or cold) objects. (All weighing must be done at ambient temperature).

3. Leave chemicals on or around balance. (Clean up your mess).

4. Write data down on loose scraps of paper. (Especially do not use paper towels!)

DO:

1. Operate all balances gently.

2. Know the name and function of all the controls.

3. Write all data in your notebook.

II. Use of the Analytical Balance

The following work is designed to acquaint you with your balance, to reveal malfunctions in the balance that may occur and to introduce you to simple weighing techniques. It is very important to perform this introductory work properly and carefully. This will give you confidence in both the equipment and your experimental techniques.

1. The laboratory instructor will demonstrate balance operations and how to handle objects for weighing. When you first turn the analytical balance on, the microprocessor will go through a self-check procedure. If the balance indicates a problem e.g. "CH 3", tell the instructor. Report any poorly functioning balance to the laboratory instructor immediately. Make sure that you learn how to calibrate the balance. It is important to calibrate the balance before measuring mass if high accuracy is desired.

2. Select the balance you will use throughout the course. Record the balance number in your notebook and sign up on the balance sign-up sheet in the balance room. It hopefully will not be necessary to have more than <u>two</u> persons assigned to each balance during any one lab period.

3. Practice weighing a solid object. Select one of the brass samples and weigh it to the nearest ± 0.0001 g. Repeat the weighing operation three times, so you can estimate the precision, and record the results on your "Summary Data Page."

4. Practice weighing a powdered sample. Fill a clean and dry weighing bottle about 1/4 full with sodium chloride. Transfer 0.50 ± 0.01 g of the NaCl into a clean, dry and previously weighed 50 or 100 mL beaker. Calculate the weight of NaCl transferred by addition and by subtraction. See the example Summary Data Page for typical calculations. While both the subtraction and addition methods can be used for this class, in many cases there are advantages to weighing by subtraction (also called weighing by difference). If you are weighing by addition into a wet flask or if the flask you are weighing into is heavy, there will be greater errors in weighing by addition. For this reason, the subtraction method of weighing needs to be learned by students. Powdered samples should be transferred directly out of weigh vials without using spatulas or weigh paper.

III. Use of the Top-Loading Balance

The top-loading balances are to be used when the full precision of an analytical balance is not required. Typical uses are weighing out large quantities of samples for drying and weighing the water during the buret calibration procedures. The lab instructor will demonstrate the operation of the top loading balance.

SUMMARY DATA PAGE
ANALYTICAL BALANCE

SAMPLE

Name	Locker Number
Section number	

PRACTICE WEIGHING

Object Weighed: brass solid Date of Weighing: 2/14/09

OBSERVED WEIGHT

Trial 1	Trial 2	Trial 3
12.3652 g	12.3651	12.3654

CALCULATIONS

Mean 12.3652 g
Standard Deviation ±0.0002 g
Rel. Std. Dev. ±0.02 ppt

WEIGHING A SAMPLE (pure NaCl)

	Trial 1	Trial 2
Wt. of beaker + NaCl	36.4583g	
Wt. of beaker	35.9470g	
Wt. of NaCl by addition	0.5113g	
Wt. of weighing bottle + NaCl	15.4967g	
Wt. of weighing bottle + remaining NaCl	14.9852g	
Wt. of NaCl by subtraction	0.5115g	
Date of Weighings	2/16/09	

Difference: Weight by addition - weight by subtraction (mg)

Trial One -0.2mg

Trial Two _____

Cleaning, Calibration and Use of Volumetric Glassware

There are three types of glassware that are routinely used for quantitative analysis and are calibrated to contain or deliver precise volumes of liquids. They are volumetric flasks, volumetric pipets, and the buret. All must be scrupulously clean to function properly. Also, a fairly high level of technical skill is required to use the pipet and buret to their maximum precision and accuracy.

The purpose of this exercise is to develop your technique in the use of volumetric glassware and to calibrate the 25 mL class A pipet and the 50 mL class A buret. The instructor will discuss and demonstrate the proper use of this equipment in a lab lecture. Class A glassware has a rated precision of ±0.1%.

Pre-Lab Calculation 1: Calculate the relative percent difference between 24.95 and 25.01 mL

I. Cleaning glassware

To deliver the correct volumes, glassware must be free of any dust, dirt, chemicals, and particularly any oils or greases. When glassware is properly cleaned, water will drain slowly and completely, leaving no droplets adhering to the walls. There are three approaches recommended for cleaning glassware. The method and materials used depend on the type of glassware and how dirty it is. The three approaches that should be used as necessary, in order are:

1. Use of soap, hot water and suitable brush

2. Use of NOCHROMIX cleaning solution

3. Use of alcoholic KOH cleaning solution

II. Cleaning Procedures

1. The buret: The buret is easily cleaned with soap, hot water and a buret brush. Merely pass the brush back and forth through the buret several times with soap and water. The Teflon stopcock (and tip) may be removed to facilitate this procedure. Rinse thoroughly with tap water then deionized water. Fill the buret with room temperature deionized water (set out at least a liter in advance) and drain water through the tip quickly to remove any air bubbles.

   Be sure to thoroughly dry the outside of the buret, including the stopcock and tip before testing for leaks! It is useful to adjust the meniscus so that it is between 0.00 and 1.00 mL on the scale. Carefully read the position of the meniscus to ±0.01 mL and write the value down. Read the position of the meniscus again after 10-15 minutes. The position should be exactly the same (within ±0.01 mL) if there was no leakage!

After the buret has stood for 10-15 minutes, carefully check the region where the glass tip fits into the Teflon stopcock. Any air bubbles indicates a leak. If there is any leakage, notify the instructor. Open the stopcock and let the buret drain to the 50 mL mark. Check for any droplets adhering to the walls of the buret. Adhering droplets indicate the buret is not thoroughly clean. Clean again if necessary.

2. Volumetric flasks: Generally, only the long neck of a volumetric flask needs to be carefully cleaned. This can be done with a test tube brush and soap and water. Always rinse with DI water before use or storage. The instructor will show what a dirty volumetric flask looks like for you to decide if additional cleaning (beyond soap and water) is needed.

3. Volumetric pipets: Volumetric pipets cannot be cleaned with a brush. Use one or more of the following methods:

a. Connect a length of rubber tubing to the top of the pipet. Connect the other end of the tubing to the vacuum connector of a water aspirator. Turn on the aspirator and slowly draw a solution of hot, soapy water into the pipet. Rinse thoroughly and check for drainage.

b. Attach a pipet bulb to one end of the pipet and fill the pipet to just past the mark with NOCHROMIX. Be careful not to suck the NOCHROMIX into the pipet bulb. Leave the bulb in place so the solution will remain in the pipet for 2-3 minutes. You should leave the pipet in the NOCHROMIX bottle during this time. Drain the NOCHROMIX back into the bottle (cleaning solutions can be used many times). Remove the pipet with a beaker under it to avoid dripping NOCHROMIX onto the floor, and rinse the outside and inside of the pipet with deionized water.

c. Use the exact same procedures as (b), but use the alcohol-KOH mixture (alcoholic KOH) solution instead of NOCHROMIX. Be careful not to place the tip of the pipet in solids which tend to form at the bottom of the alcoholic KOH bottle.

SAFETY NOTE!! NOCHROMIX contains concentrated H_2SO_4 and alcoholic KOH contains a high concentration of KOH. Both solutions can cause severe damage to eyes and skin. It is critical that you wear proper protective items (e.g. goggles, gloves and proper shoes) when using these materials. If you spill either of these solutions on yourself, rinse thoroughly with water and notify the instructor. These solutions will only be available at the beginning of the semester.

III. Use of volumetric glassware

Volumetric glassware must be used properly if the ultimate precision and accuracy are to be attained. This takes practice and patience. The lab instructor will discuss and demonstrate the procedures for using the pipet, the buret and volumetric flasks.

IV. Calibration of volumetric glassware

In this exercise you will calibrate your 50.00 mL buret and the 25 mL volumetric pipet. The volumetric flasks need not be calibrated. The calibration exercise fulfills two purposes: (1) it serves as a measure of your proficiency in the use and reading of the pipet and buret, and (2) allows you to determine the true volume delivered by these devices. In general, the procedure is a measured volume of deionized water is collected in a stoppered flask and weighed on the analytical balances to the nearest 1 mg (0.001 g) for the pipet or top loading balance to the nearest 10 mg for the buret. The "true" volume of this water is calculated by one of two methods (see below). A correction factor (ΔV) is computed by

$$\Delta V = V_{true} - V_{nominal} = \text{correction factor}$$

$V_{nominal}$ is the apparent volume, which is the volume read from the calibration marks (buret) or the nominal volume stamped on the glassware (pipet and volumetric flask). The correction factor must be <u>added</u> to all subsequent nominal volumes to obtain the true volume.

To calculate the true volume of water from its mass, it is necessary to correct for the buoyancy error and the variation of density with temperature. The buoyancy error occurs whenever the density of the object being weighed (water) differs significantly from the balance weight's density (stainless steel).

There are two ways to determine the true volume of the water:

Method 1, a two-step process:

Step 1. Correction for buoyancy.

The correct mass of an object (water in this case), m_1, is calculated from the apparent mass, m_2, by:

$$m_1 = m_2 + m_2[(d_{air}/d_1) - (d_{air}/d_2)] \qquad (1)$$

Where

d_{air} = density of air = 0.0012 g/mL
d_1 = density of object = 1.0 g/mL for water (use exact density found in tables on bulletin board or in text)
d_2 = density of balance weights = 7.8 g/mL for stainless steel

Once the correct mass is found, the true volume is calculated in Step 2.

Step 2. Conversion from mass to volume.

$$V_{true} = (\text{correct mass})/(\text{density of water}) \qquad (2)$$

A chart showing the density of water at various temperatures is posted in the laboratory. Get the instructor to show you how to read the chart.

Method 2, a shortcut, one-step method:

A table on page 38 of the course textbook lists a number of "factors" that when multiplied by the apparent mass of water measured gives the true volume directly. This factor varies with temperature and corrects for buoyancy and density. The factor has units of mL/g so:

V_{true} = factor*(m_2)

There are two columns of factors. Use the one with the heading "At temperature shown[a]".

Method 2 is OK for our work. Method 1 can only be used when temperatures are controlled to at least ±0.1 °C. This is generally not true in our lab.

1. The 25 mL volumetric pipet:

   a. Weigh an Erlenmeyer flask with rubber stopper to ±1 mg on the analytical balance.

   b. Pipet 25 mL deionized water into the flask using the pipet as instructed.

   c. Reweigh the stoppered flask and obtain the apparent weight of the water by difference.

   d. Calculate the true volume of the pipet.

   e. Repeat this procedure until you can repeatedly deliver the same volume ± 0.02 mL (which is the same as ±0.02 g). You do not have to empty the flask between measurements unless it overfills or exceeds the maximum balance weight.

   f. Calculate the average true volume delivered by your pipet based on at least 3 consecutive measurements. Record this value as your pipet volume in your notebook. Note that since the volumetric pipet delivers a single volume, it is not necessary to calculate a correction factor for the pipet.

2. The buret:

   a. Fill the buret above the top mark with DI water.

   b. Open the stopcock fully for a few seconds to flush out any air in the tip. It is critical that no air be left in the tip as it may come out during use and cause a large error in the dispensed volume.

c. Refill if necessary then slowly bring the meniscus down to just below the 0.00 mL mark. Remove any hanging droplets from the tip by touching the droplets to a waste container. (Note: It is not proper to adjust the meniscus to the 0.00 mL mark.)

d. Read the volume to the nearest ± 0.01 mL and record the value.

e. Dispense approximately 10 mL of water into a previously weighed flask (top-loading balances can be used for the buret calibration). The flow of water from the buret should be adjusted to about 1 to 2 drops per second. If the flow rate is too fast the buret will not drain reproducibly.

f. Read the volume to ± 0.01 mL and record.

g. Reweigh the flask and calculate the true volume delivered by the buret. The correction factor for the 10 mL region of the buret is calculated as:

$$\Delta V_{10\ mL} = V_{true} - V_{measured}$$

h. Refill the buret to between the 0.00 and 1.00 mL marks and dispense approximately 20 mL into the flask. Calculate ΔV for 20 mL. Repeat this procedure for 30, 40, and 50 mL volumes. In order to dispense 20 mL, fill the buret to 0-1 mL and dispense 20 mL. DO NOT simply dispense from 10 mL to 30 mL. Repeat the whole process at approximately the same volumes. No ΔV values should be larger than 0.20 mL. If your correction factor(s) are larger than this, see the instructor. Note that correction factors (ΔV's) should be reproducible. ΔV's taken at the same volume increment should not differ by more than ±0.04 mL. You must keep making measurements until consecutive ΔV values agree to within ±0.04 mL. Calculate an average ΔV for each volume increment using your most reproducible values.

V. Results

Plot a "buret calibration graph" by plotting ΔV vs. buret volume. Use average ΔV values and nominal buret volumes (10, 20 mL etc.).

Use a spreadsheet program of your choice to make the plot. Refer to EXERCISE 3 in the Computer Use section for help. Turn one in to the instructor as part of the laboratory report. Once this is approved, attach it in the laboratory notebook.

Data Summary Page
CALIBRATION OF 25 mL PIPET

Name	Locker Number
Section number	

MEASUREMENT	Trial 1	Trial 2	Trial 3
Wt. of Erlenmeyer + Water			
Wt. of Erlenmeyer			
Apparent weight of water			
True pipet volume			
Date of measurements			

Ambient (water) temperature _____ ^{o}C

Volume of 1 g of water at ambient temperature _____ mL

Average pipet volume delivered _____ mL

Standard deviation of volume ± _____ mL

Relative standard deviation of volume ± _____ ppt

Pipet Calibration Report

Name _____

Lab Section _____

Submit the average volume delivered for the twenty-five mL volumetric pipet using the last three or four consecutive pipet volume values. Also include the standard deviation, the relative standard deviation (in parts per thousand), and the 95% confidence interval for these values.

Trial

1	**2**	**3**	**4**
_____	_____	_____	_____

Average volume (mL) _____

Standard Deviation (mL) _____

Relative Standard Deviation (ppt) _____

95% Confidence Interval _____ ± _____

SAMPLE CALCULATION

Continue on reverse side if necessary.

SUMMARY DATA PAGE - CALIBRATION OF 50 mL BURET

Name	Locker Number
Section number	

TRIAL ONE

Ambient Water Temperature (°C) _____

Volume of 1 g of water at ambient temperature _____mL

Date of work					
Range on the Buret	0-10	0-20	0-30	0-40	0-50
Final Buret Reading					
Initial Buret Reading					
Apparent Volume Delivered					
Weight of Flask+Stopper+Water					
Weight of Flask+Stopper					
Apparent Weight of Water					
True Volume of Water Delivered					
Correction Factor (mL)					

TRIAL TWO

Date of work					
Range on the Buret	0-10	0-20	0-30	0-40	0-50
Final Buret Reading					
Initial Buret Reading					
Apparent Volume Delivered					
Weight of Flask+Stopper+Water					
Weight of Flask+Stopper					
Apparent Weight of Water					
True Volume of Water Delivered					
Correction Factor (mL)					
Average Correction Factor					

Note: A slightly different table may be desired if the two step correction method is used.