Experiment 1

INTRODUCTION TO THE ANALYTICAL BALANCE AND VOLUMETRIC GLASSWARE

2 lab periods

Reading: Chapter 2, Quantitative Chemical Analysis, 7th or 8th Edition, Daniel C. Harris and CHEM 253 Quantitative Analysis Laboratory Experiments, 7th Edition, pg 4-11.

Objective
This lab will introduce the basic laboratory equipment and skills needed for accurate and precise quantitative analysis.

Schedule
Lab 1   Practice using the analytical balance and clean your glassware. A brief soaking in warm detergent solution should be enough to remove grease and dirt. If not, consult your TA. Become familiar with your glassware and begin calibrating it.
Lab 2   Calibrate your glassware. During this lab period you will need to check out one or more items from Lab Services. Sometimes long lines can develop at Lab Services, especially in the afternoon sections. If there is a long line, start working on the parts of the experiment you can do without the checked out equipment. Usually the lines are much shorter once the initial "rush" dies down.

Use of the Analytical Balance
        The ability to determine masses accurately is fundamental not only to analytical chemistry but to all of modern science. All measurements that we will perform in this course relate either directly or indirectly to weighing using an electronic analytical balance. The analytical balances in Chem 253 can be used to obtain four or five digits to the right of the decimal point. They have a precision of ± 0.1 mg. These are delicate and precise instruments, which, if kept scrupulously clean and handled correctly, will continue to yield reliable data during the course of their usage. It is important that you become well acquainted with your balance. You should be confident that the balance is working properly and that your technique is correct before you proceed with any experiment. You should also read the section in your text on the analytical balance (Section 2.3 in *Quantitative Chemical Analysis, 8th Edition*, pp. 31-34).

1. *Introduction to the balance:* Attend a demonstration by your instructor on how to operate the balance and how to handle objects for weighing. After this demonstration, familiarize yourself with the controls on the balance and learn to read the optical scale to the nearest 0.1 mg (0.0001 g). Zero the optical scale with the pan clean and empty.

    Report any defects or poorly functioning controls to your TA immediately; do not try to make any adjustments until you have received specific instructions. Check the level of the balance. If the balance is out of level, please report it to the TA. Do not try to adjust the level by yourself.

    The floor and pan of the balance should be scrupulously clean. The balance doors should be closed except when you are loading your sample.

    A further note of caution is that all objects or samples to be weighed must be brought to thermal equilibrium with the room (ambient temperature) before a weighing can be

attempted. If you are unsure whether your sample is at ambient temperature, put the sample on the balance and observe whether the reading changes over a two-minute period. Temperature gradients within the balance can cause convection currents and a hot sample will lead to buoyancy errors, both of which can degrade your results.

2. *Weighing out samples:* Weighing by difference is the most accurate method we can use to quantitatively determine the mass of a solid sample. This procedure involves repetitive weighings of a weighing bottle containing a quantity of solid reagent. As the solid is <u>removed</u> to another vessel, the mass of the weighing bottle plus contents decreases. Upon reweighing, a lower mass is found. The difference in the two masses represents the mass of solid reagent transferred to the vessel. Hence, the phrase "weighing by difference". In this experiment you will use common table salt (NaCl) to practice weighing by difference. The goal is to weigh out three 0.7 g aliquots of NaCl as accurately as possible.

Practice weighing by difference

1. Place a*pproximately* 3 g of NaCl (plain table salt) in a clean, dry glass weighing bottle.

2. Take this into the weighing room, along with your lab notebook and three Erlenmeyer flasks.

3. Each student will be assigned a balance. Find the balance assigned to you.

3. Use a piece of folded paper (clean, dry and lint-free) to handle the weighing bottle. This is to avoid leaving fingerprints or oils on the bottle and changing the mass of the bottle. Place the weighing bottle carefully on the balance pan.

The decision whether or not to leave the top on the weighing bottle is usually determined by the hygroscopic (ability to adsorb atmospheric water vapor) nature of the solid reagent. If your reagent is hygroscopic, it is best to minimize contact with water vapor by keeping the stopper on the weighing bottle as much as possible. If your reagent is not appreciably hygroscopic, the stopper may be left off for the duration of the weighing process. NaCl is not hygroscopic.

4. Record the mass of the weighing bottle in your notebook.

5. Remove the weighing bottle and transfer some of the NaCl to the beaker or flask in which you will perform the determination.

The transfer must be quantitative. Make certain that all the NaCl removed from the weighing bottle ends up in the beaker or flask. Do not use a funnel or piece of paper to effect the transfer, as some NaCl may remain on the surface. Perform a direct transfer of the NaCl from the weighing bottle to the beaker or flask by "rolling" the weighing bottle on its side over the mouth of the vessel. You may use a clean, dry spatula to help in the transfer, as long as you make sure that no NaCl remains on the spatula (rinsing, etc.).

6. Replace the weighing bottle on the balance pan and record the new mass. Subtract this new, smaller, mass from the previous mass. Your sample should weigh somewhere in the range of 0.6800 to 0.7200 g. Don't worry if the mass is *slightly* over or under these values.

You may find it hard at first to estimate the point at which you have transferred ~0.7 g into your flask or beaker. You might have to try the transfer several times until you get it right. If you overshoot, don't transfer any reagent back into the weighing bottle. Simply discard the solid reagent in the proper manner (see the TA for instructions), rinse out the vessel, and begin the

weighing and transfer process over again. Once you have an appropriate amount of reagent transferred, use this amount as a visual guide to estimate subsequent samples.

7. Repeat these steps for each of your three samples. Record your masses in your notebook as follows. Make sure to use proper mass units.
    Initial (larger) mass (g)
    - Final (smaller) mass (g)
    Difference (g)

8. You should be able to accurately weigh three samples in ten minutes. Since there will be more than one student assigned to each balance, access to the balances is on a first-come, first-served basis.

9. Clean up your balance station.

Calibration of Volumetric Glassware
Quantitative analysis is a very precise science and can only be completed successfully with exact measurements and data. Glassware used in quantitative experiments is of the highest grade, but it is not perfect. Therefore, every piece of volumetric glassware must be calibrated in order to allow for the slight variations present in every piece of glassware.

Calibration of a 50.00 mL Buret
Check out a buret from Lab Services. This buret is the one you will use throughout the semester.

1. Fill the buret to just past the 0.00 mL mark with deionized (DI) water. Force any bubbles out of the tip of the buret.

Be sure the liquid in the buret drains down the walls of the column without sticking to the sides and forming droplets. If these droplets do form, the buret is dirty and needs to be cleaned. Record the level of water in the buret. Remember, when reading the buret, always use the bottom of the meniscus to take your measurement. Estimate your reading to the nearest 0.01 mL.

2. Touch the tip of the buret to the inside rim of a beaker to get rid of any suspended water. Wait approximately one minute before reading the buret, in case the level drops due to leakage. If you notice any leaks in the stopcock area, tighten the stopcock or obtain a different buret.

3. Make a table in your lab notebook like the example given below (Table 1). You will use it to record your data.

4. Weigh a 60 mL plastic bottle on the *top-loading balance*. The top loading balances used in Chem 253 have a precision of ±1 mg, which is sufficient for our purposes here. Record this weight in your table. You will use this to determine the mass of water delivered by the buret. Remember, hold the bottle with a paper towel or tissue, in order to avoid leaving fingerprint residue, which may affect your results.

5. Observe the level of the water in your buret and record it to the nearest 0.01 mL in your table under Initial Reading.

6. Drain approximately 10.00 mL into the plastic bottle that you have weighed. Tap the tip of the buret on the inside edge of the bottle to remove any suspended water. Replace the cap on the bottle as soon as you're finished in order to prevent evaporation of the water. Allow about a minute to pass before measuring the new level of water in the buret. This allows the water time to drain down the walls of the buret column. Finally, read the water level in the buret to the nearest 0.01 mL and record it in your table under Final Reading.

7. Weigh the bottle with the water in it on the top loading balance. Subtract the original weight of the bottle from the new weight. Record the data with three places after the decimal, i.e.: 10.006 g. Record this result in your table under "mass of the bottle plus water".

8. Repeat the procedure at increments of 10.00 mL until you have made five measurements. You should have delivered ~ 50 mL from your buret at this point.

9. Calculate the volume delivered by the buret for each measurement. (Subtract the initial buret reading from the final buret reading, as shown in the example.)

10. Use your measured masses to calculate the volume of water *actually* delivered by the buret (true volume). Measure the temperature of the water in degrees Celsius to the nearest 0.1° C and record it in your notebook. Then find the corresponding density of water at that temperature in Table 2. Use this density value in your calculation. An example of this calculation is shown for Trial 1 at 21.5° Celsius.

11. In order to determine the correction factor, subtract the final reading from the true volume. An example of this is show for Trial 1 below.

12. Make a calibration curve for your buret like the one shown in Figure 1 below.

Table 1   Calibration of a 50.00 mL buret, Trial 1

	10 mL	20 mL	30 ml	40 mL	50 mL
Mass of the bottle (g)	30.000				
Mass of the bottle plus water (g)	39.895	49.876	59.856	69.883	79.661
Mass of water (g)	9.895	19.876	29.856	39.883	49.661
Final Reading, buret (mL)	10.22	20.23	30.23	40.24	50.00
Initial reading, buret (mL)	0.23	10.22	20.23	30.23	40.24
Volume delivered, buret (mL)	9.99	10.01	10.00	10.01	9.76
Total volume delivered, buret (mL)	9.99	20.00	30.00	40.01	49.77
True volume calculated from mass of water (mL)	9.91(58)	19.91(82)	29.91(92)	39.96(77)	49.76(64)
Correction (mL)	-0.07	-0.08	-0.08	-0.04	-0.01

Example calculations
Volume delivered = final volume – initial volume
        10.22 mL – 0.23 mL = 9.99 mL
True volume = mass /density
        9.895 g × (1 mL / 0.99789 g) = 9.916 mL (at 21.5°C)
Correction factor, 10 mL = true volume – volume delivered
        9.915(8) mL – 9.99 mL = -0.074

Making the calibration curve
Calculate a correction factor for each volume. Plot the correction (mL) vs total volume delivered (mL) using Excel or a similar plotting program. Make sure your plot has a title, labeled axes, and units.
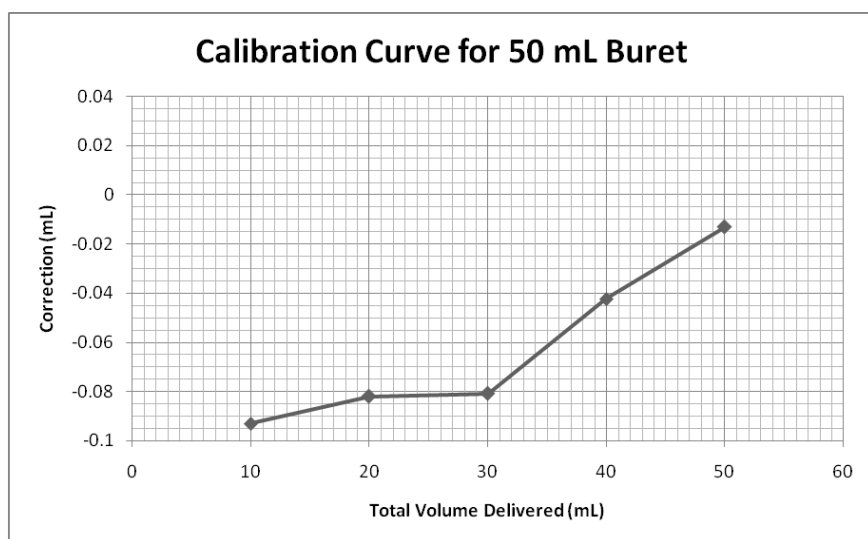

Calibration Curve for 50 mL Buret

Figure 1
How to use the calibration curve:
The buret used to acquire the above data doesn't really deliver the exact volume that we read from the buret. The true volume delivered (calculated from the mass) is consistently less than it should be. However, it is not convenient to always weigh out whatever analyte is being delivered from the buret. Therefore, we use the calibration curve to correct the volumes delivered from the buret. The tolerance of a Class A 50 ml buret is ±0.05 mL. If your buret is far out of this range, you'll want to trade it in for a new one from Lab Services. Remember to calibrate your new buret, too.

How to apply the correction:
Amparo used the above buret to deliver 30.21 mL of a solution. She calculates the true volume delivered in the following way:
        volume read from buret + correction factor at a particular volume = corrected volume.
        30.21 mL + -0.08 mL = 30.13 mL
The corrected volume is 30.13 mL. This is the value Amparo will use in her calculations.
Always use the calibration curve when you use your buret. Be careful when acquiring the data for your calibration curve. Make sure you read the buret carefully.

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Calibration of 5, 10 and 25 mL pipets.
Calibrate your pipets in the same way as for the buret. Make three separate measurements for each pipet. Calculate the average and the standard deviation for your data from the averages. Calculate the true volume delivered from the pipet from the mass of the water, as you did in the buret calibration procedure. Calculate a correction factor for each pipet.

Calibration of your 50 and 100 mL volumetric flasks.
Calibrate your 50 and 100 mL volumetric flasks in the following way:
Place the empty volumetric flask on the top-loading balance and write down the weight of the flask. Remove the flask from the balance. Then, carefully add DI water just until the bottom of the meniscus rests on the top of the mark on the neck of the flask. Reweigh. Discard the water and refill the flask. Repeat three times. Calculate the mean and standard deviation, the true volume, and the correction factor.

Table 1.2   Density of water as a function of temperature

T, °C	Density g/mL	T, °C	Density g/mL	T, °C	Density g/mL	T, °C	Density g/mL
19.0	0.99842	21.0	0.99800	23.0	0.99755	25.0	0.99705
19.1	0.99840	21.1	0.99798	23.1	0.99752	25.1	0.99703
19.2	0.99838	21.2	0.99796	23.2	0.99750	25.2	0.99700
19.3	0.99836	21.3	0.99794	23.3	0.99748	25.3	0.99698
19.4	0.99834	21.4	0.99792	23.4	0.99745	25.4	0.99695
19.5	0.99832	21.5	0.99789	23.5	0.99743	25.5	0.99692
19.6	0.99830	21.6	0.99787	23.6	0.99740	25.6	0.99690
19.7	0.99828	21.7	0.99785	23.7	0.99738	25.7	0.99687
19.8	0.99826	21.8	0.99783	23.8	0.99735	25.8	0.99684
19.9	0.99823	21.9	0.99780	23.9	0.99733	25.9	0.99682
20.0	0.99821	22.0	0.99778	24.0	0.99731	26.0	0.99679
20.1	0.99819	22.1	0.99776	24.1	0.99728		
20.2	0.99817	22.2	0.99773	24.2	0.99726		
20.3	0.99815	22.3	0.99771	24.3	0.99723		
20.4	0.99813	22.4	0.99769	24.4	0.99721		
20.5	0.99811	22.5	0.99767	24.5	0.99718		
20.6	0.99809	22.6	0.99764	24.6	0.99716		
20.7	0.99807	22.7	0.99762	24.7	0.99713		
20.8	0.99805	22.8	0.99760	24.8	0.99710		
20.9	0.99802	22.9	0.99757	24.9	0.99708		

Report Sheet Experiment 1   **Name/Date** _____

Calibration of Volumetric Glassware

Experimental Purpose

Data and Calculations

1.   Calibration of a 50 mL buret

Water temperature:_____

	10 mL	20 mL	30 ml	40 mL	50 mL
Mass of the bottle (g)					
Mass of the bottle plus water (g)					
Mass of water (g)					
Final Reading, buret (mL)					
Initial reading, buret (mL)					
Volume delivered, buret (mL)					
Total volume delivered, buret (mL)					
True volume calculated from mass of water (mL)					
Correction (mL)					

Attach the graph of average correction factors vs. apparent volumes (total volume delivered) to the Report Sheets.

Calibration of pipets	5 mL	10 mL	25 mL
Mean Correction (mL)			
Mean true volume ± standard deviation (mL)			

Calibration of volumetric flasks	50 mL	100 mL
Mean Correction (mL)		
Mean true volume ± standard deviation (mL)		

Attach a copy of the sheets from your lab notebook at the end of your report.