

EXPERIMENT 1

Laboratory Techniques: Unit Operations

IMPORTANT NOTICE FOR THE CHE 226 LABORATORY

“Dry labbing” or “pencil titrating” (that is, fabricating or making up data), copying from anyone else’s lab report, plagiarism from the text or from any other source, on any portion of this lab or any other lab is academic dishonesty. The minimum penalty is failure of the course. Carefully read the section on Academic Dishonesty in the course syllabi. If you have any specific questions about academic dishonesty, ASK THE INSTRUCTOR.

RECORDING DATA

Use a printed copy of the spreadsheet provided to record your data for this experiment, and this experiment only. For all other experiments, use only your Laboratory Notebook. **Pieces of scrap paper on which you have written will be confiscated throughout the semester.**

UNKNOWNNS

The Teaching Assistant will provide you the unknowns for this laboratory: (a) A numbered aluminum slug in a glass vial and (b) A block of wood with five partially filled burette sections. **These must be returned as soon as you have completed the Part of this lab in which each is used.** If you have to repeat one of these Parts, you will be issued a new unknown for that Part.

INTRODUCTION

The purpose of this experiment is to introduce several of the tools and techniques necessary for success in this course. The techniques are considered one at a time, as though they were unit operations. You may well have performed each of these techniques in previous chemistry labs, or even in undergraduate research, and are *absolutely certain* you know how to perform them well. This is immaterial, and you’re probably wrong anyway. There’s a wise, old adage: “It isn’t the things you don’t know that get you into trouble. It’s the things you know for certain that just aren’t true.”

The Analytical Chemistry laboratory requires a substantially higher level of precision, accuracy, laboratory technique, and cleanliness than any other laboratory you have hitherto encountered. To quote the Bible:

*Thou shalt not have in thine bag divers weights, a great and a small.
Thou shalt not have in thine house divers measures, a great and a small.
But thou shalt have a perfect and just weight, a perfect and just measure shalt thou have...*
–Deuteronomy 25: 13-15

Much as a golfer studies, practices, and improves individual discrete skills of the game (the grip, the stance, the swing, driving, putting, etc.) and puts these all together when playing a round of golf, the chemist must study, practice, and improve individual laboratory techniques such as pipetting, weighing, transferring solids and solutions, and reading a burette to do a complete analysis properly.

The whole point of this experiment is to learn proper technique for individual skills first, before attempting additional labs. If you can't read a burette properly or pipet accurately or reproducibly, it is foolish to attempt a complete volumetric analysis. Therefore, if any parts of this experiment need to be redone to satisfy the various tolerance levels, **redo them as soon as possible**, and turn the report back in for re-grading. If you have done a particular Part two or three times and still have not met tolerance limits, you are obviously doing something wrong. Ask for assistance from one of the instructors.

GRADING OF EXPERIMENT #1

This experiment is graded differently from all others in the course: A student must pass each and every Part successfully to be done with the experiment. Once each Part is successfully completed, you will have earned 100 points. To ensure that these important skills have been mastered in a timely fashion, this experiment **MUST** be successfully completed and turned in by the end of your 5th scheduled laboratory period. If not, a 0 will be awarded.

There is no “partial credit” on this experiment. You will be awarded either 100 points or 0 points.

PART A: USE OF THE ANALYTICAL BALANCE

In this experiment, you will obtain the masses of five new pennies – first by weighing each penny individually, and then by weighing all five pennies at once, removing one penny at a time, and obtaining the individual masses of the pennies by difference. The pair of masses determined for a particular penny should agree to within a few tenths of a milligram. From the data, you will determine the average and median values, the standard deviation, and the relative standard deviation in the masses of the pennies.

You will then weigh an “unknown” aluminum slug, and report its mass and code number.

Procedure

After the Teaching Assistant has instructed you in the proper use of the electronic balance and you have become familiar in its use, obtain a set of pennies, an unknown aluminum slug, and a pair of tweezers from the instructor. Never handle the pennies or the slug with your fingers, always use the tweezers.

1. Go to the analytical balance that has been assigned to you. Zero it carefully. Select five pennies at random from the vial containing the pennies. Keeping track of which penny is which, weigh each penny, # 1-5, *one at a time* on your balance. Enter the masses on the data sheet provided.
2. Re-zero the balance. Place all five of the same five pennies on the balance pan, obtain the total mass and enter it on the same data sheet. Remove penny #5 from the balance, obtain the mass of the remaining four and record the mass. Repeat this process, removing one penny at a time. Obtain the individual weights by subtraction. This process is known as *weighing by difference*, which is the way almost all weighings are done in the Analytical Laboratory.
3. Now weigh the unknown aluminum slug and record its mass.
4. Perform the calculations requested and have the TA check your results.

RETURN THE VIAL OF PENNIES AND THE AL SLUG AS SOON AS YOU HAVE SUCCESSFULLY COMPLETED THIS PART OF THE EXPERIMENT.

PART B: QUANTITATIVE TRANSFERS

The following experiment is designed to provide experience in the correct use of the volumetric flask.

1. Tap a very small amount of potassium permanganate, KMnO_4 , from the stock bottle onto a piece of folded glassine paper or into a small clean beaker or a plastic weighing boat. (Note: Chemicals should **never** be placed back into stock bottles as this may contaminate the entire bottle. Avoid putting a spatula into a stock bottle. Tap out a small amount if at all possible.)
2. Tare a clean, dry 50-mL beaker on an electric balance. Add about 0.4 g of KMnO_4 to the beaker. **NOTE: NEVER transfer chemicals inside an analytical balance.**
3. Dissolve the potassium permanganate in about 20 mL of distilled water, stirring gently to avoid loss. This is nearly a saturated solution, and some care is required to dissolve the crystals completely.
4. *Quantitatively* transfer the solution into a 100-mL volumetric flask using a small funnel. To prevent the solution from running down the outside of the beaker, pour the solution down the

stirring rod, and then touch the rod to the spout of the beaker to remove the last drop. If needed, ask a TA to show you how to do this. Add more water to the beaker, stir, and repeat the procedure. Note the amount of washing required to *quantitatively* transfer the permanganate from the beaker to the flask. Finally, rinse the last portions of solution from the stirring rod into the volumetric flask with a stream of water from the wash bottle. Rinse the funnel and remove it. Carefully dilute the solution in the flask until the bottom of the meniscus is even with the graduation mark.

5. Stopper, invert, and shake the flask. Return it to the upright position, and allow the air bubble to return all the way to the top of the neck. Repeat until the solution is completely homogeneous; about 10 inversions and shakings are required. Save the solution for Part C.

PART C: TAKING AN ALIQUOT

Whenever a burette or pipet is used to deliver a measured volume of solution, the liquid it contains before a measurement must have the same composition as the solution to be dispensed. The following operations are designed to illustrate the *minimum* effort needed to ensure this.

NOTE: During this Part, collect all the permanganate solutions and rinsings in a large beaker ("slop bucket"). At the end, all the permanganate is to be disposed of in the proper, labeled waste container. **No permanganate should go down the drains.**

1. Fill a pipet with the solution of potassium permanganate and let it drain completely into a waste beaker. Wait 20-30 seconds, then touch the tip of the pipet to the side of the beaker. Draw a *small* amount of distilled water from a 50-mL beaker into the pipet, rinse, and discard the rinse solution. Do not fill the pipet completely; this is wasteful, time-consuming, and inefficient. Just draw in a small amount, tilt the pipet horizontally, and turn it to rinse the sides. Determine the minimum *number* of such rinsings required to completely remove the permanganate color from the pipet. [Hint: Look at the liquid that collects in the tip of the pipet against a white background to see if there is any coloration.] If your technique is efficient, three rinsings should suffice.
2. Fill the pipet again with permanganate and proceed as before. This time determine the minimum *volume* of rinse water required to remove the color by collecting the rinsings in a **small** graduated cylinder (less than 5 mL of rinse water is enough with efficient technique). Again, check the pipet tip as before for any coloration.
3. As a test of your aliquoting technique, ask the laboratory instructor to observe and comment on the following operation: Rinse a 10-mL pipet several times with the solution of potassium permanganate you prepared. Pipet 10 mL of the permanganate solution into a 250-mL volumetric flask. Carefully dilute the solution to volume, trying to mix the contents as little as possible. Now, mix by repeatedly inverting and shaking the flask. (Note the effort required to disperse the permanganate color uniformly through the solution.) Rinse the pipet with the solution in the volumetric flask. Pipet a 10-mL aliquot of the solution into a conical flask.

4. If the laboratory instructor considers your technique satisfactory, ask him/her to initial your report to that effect.

PART D: CALIBRATION OF A PIPET

The calibration of an analytical transfer pipet is a relatively straightforward procedure. The proper technique is readily learned with practice, care, and attention to detail. Note that this is a *manual* technique. It must be learned; it does not magically appear on the first try, not unlike learning to hit a golf ball. With some practice, however, you ought to be able to handle a pipet about as well as anyone on the face of the earth. With the exception of a simple weighing, this experiment is capable of being the most accurate and precise set of measurements you're likely to ever perform in your life.

1. Obtain the following equipment: Pipet bulb; 50-mL Erlenmeyer flask with a solid, dry cork or rubber stopper; 400-mL beaker of distilled water equilibrated to room temperature; and a thermometer. Thermometers are common-use equipment and usually available on the side bench. **Return your thermometer as soon as you are done with this Part of the Experiment.**
2. Clean a 10-mL pipet, and ask the laboratory instructor to verify that your pipet is clean. Cleaning is usually accomplished by drawing some warm soapy water into the pipet bulb, wetting the sides, and "shaking the soap" inside the pipet. If that doesn't work, soak the entire pipet in soapy water overnight or longer. You must clean pipets, burettes, and other pieces of volumetric glassware, so that no droplets of reagent remain on the internal surfaces when they are drained. This is very important for accurate and reproducible results. If reagent gets "hung up" inside a pipet, you obviously cannot deliver the nominally stated volume. You should have already cleaned your pipet properly during the check-in period.
3. Weigh the flask and stopper and record the mass to the nearest 0.1 mg. Do not touch the flask with your fingers after this weighing. Use tongs or wear gloves.
4. Read and record the temperature of the water.
5. Pipet 10.00 mL of the distilled water into the flask using the technique described in the text. Restopper the flask, weigh it, and record the mass of the stoppered flask plus the water.
6. Add a second pipet of water to the flask, *without pouring out the 1st aliquot*, removing the stopper just before the addition. Re-stopper, weigh, and record the weight. Repeat the entire procedure for a *minimum* of four readings that agree to within a *total range* of less than about 0.02 g for all the readings. [If you have what you consider 3 "good" replicates and one that seems "bad", try the *Q*-test to see if the outlying value can be rejected.] If your values seem very non-reproducible, throw out all your data and do another full set of 4 minimum, paying closer attention to what you're doing. If these still do not reproduce well, there is something clearly wrong with your technique. Seek assistance from one of the instructors.

7. Correct the *apparent masses* of the aliquots for the buoyancy effect of atmospheric air to get the *true masses* as described below. Then calculate the *true volume* of the pipet using the density of water at the temperature(s) of each aliquot.
8. Report the average “true volume” of your pipet and the associated standard deviation of your values.

Correction for Buoyancy

A buoyancy error will affect data if the density of the object being weighed differs significantly from that of the standard weights (inside the balance for an electric balance). This error is due to the difference in the buoyant force exerted by the medium (air) on the object weighed and on the weights themselves. The correction is made using:

$$m_{corrected} = m_{observed} + m_{observed} [(d_{air}/d_{object}) - (d_{air}/d_{weights})]$$

where m is the mass in grams of the corrected and initially observed values of the object weighed (in this case, distilled water), and d is the density in g/cm^3 of air, the object, and the weights.

For all but the most exacting work, this correction is negligible for solids and liquids having a density of $2 \text{ g}/\text{cm}^3$ or greater. Correction for buoyancy is required only for measurements that require the highest accuracy, for gases, or for low-density solids and liquids.

The density of the weights used in electronic single-pan balances ranges from 7.8 to $8.4 \text{ g}/\text{cm}^3$, depending on the composition of the weights. Using $d_{weights} = 8 \text{ g}/\text{cm}^3$ is adequate for most purposes. The $d_{air} = 0.0012 \text{ g}/\text{cm}^3$ at room temperature and pressure.

PART E: READING BURETTE SECTIONS

1. Obtain a set of five “burette sections” from the Teaching Assistant. The sections are normally stored upside down so that the inside surface of the part of the section where you will take your reading will remain wetted and drain well.
2. Invert each section and tap the section lightly to remove any solvent that might remain in the sealed tip and let it drain for at least 20-30 sec. Record the number and reading of each burette section on the form provided. Use a burette reading card to estimate the readings to the nearest 0.01 mL. [A burette card is easily made by making a very heavy, black horizontal rectangle in the center of a 3 x 5” file card.
3. Repeat for a second set of burette sections.]

PART F: USE AND PROPER READING OF YOUR BURETTE

1. Over-fill the burette with distilled water. Make sure that there are no air bubbles trapped in the tip. If so, rapidly open and close the valve several times until the bubbles are flushed out. (Why is this necessary?)
2. Drain the burette down to someplace between 0 and 1 mL. Do not try to bring the burette to *exactly* 0.00 mL, as this is a waste of time. The “zero” reading on all but an automatic burette is always some finite non-zero reading.
3. Wait at least 20-30 seconds before taking the initial or “zero” reading. Why? Take the initial or “zero” reading and all other burette readings using a burette reading card.
4. Now let about 5 mL run into a 250-mL Erlenmeyer flask. Wait at least 30 seconds and take the “final reading”. (The amount of solution in the Erlenmeyer flask is equal to the final reading minus the “zero” reading.) Write down the final reading on the form provided, and then ask your friendly Teaching Assistant to take the final reading. Compare the two readings. They should agree within 0.01 mL. Do they? Notice that the final digit in the burette reading is the estimation of the distance between two marks on the burette.
5. Refill the burette, and take a new zero reading. Now add 30 drops to the Erlenmeyer flask, and take the final reading. Calculate the average volume of one drop, then repeat this except with 40 drops and calculate the average volume of a drop. Record these results and compare them. Now, practice adding “half-drops” to the flask. Calculate the average volume of your “half-drops”. In your volumetric unknowns, you will want to try to get half-drop end points for good precision.

HAZARDOUS WASTE DISPOSAL

Dispose of ALL solid permanganate, permanganate solutions, and washes containing permanganate into the proper, labeled Hazardous Waste Container in the hoods. If you are unsure about the container, ASK.

TEXT REFERENCE

D. A. Skoog, D. M. West, F. J. Holler, and S. R. Crouch, *Analytical Chemistry: An Introduction*, 7th ed. Chapter 2, pp. 21-59.

NOTE

The file of the spreadsheet on pp. 10-11 is designed to automatically perform needed calculations on the data entered. See the CHE 226 Website or ask for a file. Alternatively, you can simply perform the calculations yourself and write the results in. It is probably a good idea to use the file and also do the calculations manually as calculation practice with real data. The three zeros

already showing for n , the number of replicates, are placed there to automatically count the number of replicate data entered in that Part of the Experiment. As you enter data into the file, they will change.

Revised August 24, 2004

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THE DENSITY OF WATER IN g/cm³ NEAR ROOM TEMPERATURE

°C	.0	.1	.2	.3	.4	.5	.6	.7	.8	.9
18	0.998595	576	558	539	520	501	482	463	444	424
19	405	385	365	345	325	305	285	265	244	224
20	0.998203	183	162	141	120	099	078	056	035	013
21	0.997992	970	948	926	904	882	860	837	815	792
22	770	747	724	701	678	655	632	608	585	561
23	538	514	490	466	442	418	394	369	345	320
24	296	271	246	221	196	171	146	120	095	069
25	0.997044	018	0.996992	967	941	914	888	862	836	809
26	0.996783	756	729	703	676	649	621	594	567	540
27	512	485	457	429	401	373	345	317	289	261
28	232	204	175	147	118	089	060	031	002	0.995973
29	0.995944	914	885	855	826	796	766	736	706	676
30	0.995646	616	585	555	525	494	464	433	402	371