EXPERIMENT 1

Laboratory Techniques: Unit Operations

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 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