Chemistry 151/151L Quantitative Analytical Chemistry

Check-In and Cleaning of Glassware

I. Required Reading

Skillful and knowledgeable use of analytical equipment makes a huge positive difference in your results, so you should always acquaint yourself with the accepted procedures BEFORE entering the lab. In addition to reading this project description and answering questions in the prelab section, you should read the following chapters of the course textbook (Fundamentals of Analytical Chemistry (8th edition) by D.A. Skoog, D.M. West, F.J. Holler, S.R. Crouch): Chapters 2, 5, 6, 7-A. If you do not have experience with Excel calculations, you should read Chapter 3 as well. Review Chapter 4 if you have forgotten General Chemistry.

II. Check In / Overview

1. Your TA will conduct a safety/information session at the beginning of the lab. Be sure to familiarize yourself with the lab layout, location of safety equipment, safety showers, etc. Write down the contact information for your TA. Hand in the safety contracts to your TA at the beginning of the lab.

2. You will be assigned a storage drawer and a set of glassware. To get your locker, write down the range of locker numbers available on your bench and go to the stockroom with your UCI ID to sign out a locker number and get a lock code. Once you occupy the locker, label it with labeling tape as follows:

   Your name, your section, FALL 2009

Please do not occupy the locker if it is already in use by a student from an earlier lab session (it will have a similar label).

3. Check the equipment in your drawer against a standard list, acquire any missing items from the stockroom, and check your glassware for any cracks. Exchange the broken glassware with the stockroom if needed. At the end of the quarter you will be required to demonstrate that all your equipment is accounted for and clean during the check-out.

4. In analytical chemistry, measurement errors are just as important (if not more important) as the measurement result itself. Every report you will be writing for this course will include a detailed error analysis. Your Chem 151L course instructor will provide an overview of the error analysis during the discussions. Errors will be also covered in Chem 151 to some extent. In addition, your TA will give you an overview of error analysis during the first lab.

III. Cleaning Your Glassware

You should thoroughly clean ALL glassware items in your drawer, even if they look clean. This is especially critical for volumetric glassware such as pipettes, burettes, and volumetric flasks. Rinse your glassware with tap water several times and then with de-ionized water (DI water, available at the sink) a couple of times. Fill your wash bottle with nanopure water so you can use it to rinse/coat your glassware. Do not needlessly waste nanopure water because it takes a while to make. Check the cleanliness of the glass by observing the manner in which water drains from the
glass surface. Water will drain smoothly from a clean glass surface. Breaking of water film into streaks, or even worse, into small drops is the sign of a greasy surface.

Before cleaning the burette verify that it even works. Fill the burette with nanopure water for at least 10 minutes and observe for leaks near the stopcock. Next, drain the water making sure that it flows smoothly and continuously through the stopcock. There is no point in cleaning the burette only to discover later that it is leaking or has a broken valve.

There should be dropper bottles filled with cleaning solution in the lab which can be used to clean the glassware. The cleaning solution is called Nochromix. It contains concentrated \( \text{H}_2\text{SO}_4 \) and a powerful oxidizer, so treat it with great caution.

**Wear your goggles, long sleeve attire with a lab apron and gloves when working with Nochromix. Do the cleaning in a fume hood and lower the sash to protect your upper body against accidental droplets. Do not lean onto the hood surfaces to prevent your clothing from being soaked with accidentally spilled Nochromix.**

When cleaning with Nochromix, coat the glass completely and let it sit for several minutes. Drain this used but undiluted Nochromix into a small beaker. Do not dispose of the Nochromix solution; use the same solution on different glassware items. Nochromix can be reused many times; using it just once is very wasteful. When you are done treating all glassware with Nochromix, pour your used but undiluted Nochromix into a glass jar labeled “recycled / used Nochromix” located in the fume hood designated for neutralization. When you are ready to rinse your acid coated glassware, first put a tea spoon of sodium bicarbonate, \( \text{NaHCO}_3 \), into a large beaker, and add 50 mL of tap water. Rinse your glassware many times with small quantities of DI water, and let the rinse water drip into the beaker. Do not place bicarbonate in your glassware to neutralize the acid; this must be done by extensive rinsing instead. Rinsing should remove all acid from your glassware. Add more sodium bicarbonate to your beaker as needed so that there is always some amount of the white \( \text{NaHCO}_3 \) solid at the bottom of your beaker.

Recheck the cleanliness of the glass by observing the manner in which water drains from the glass surface. If you cannot get it clean with one Nochromix treatment, do the following: 1) Clean the glassware using baking soda and an appropriate brush; 2) Rinse it thoroughly with tap water to get rid of remaining soda; 3) repeat the Nochromix treatment. If even this does not help, please exchange it for another piece of glassware from the stockroom. That will also need to be cleaned.

One of the fume hoods will be reserved for neutralizing your acidic rinse water. Take your beaker to this fume hood, and place it inside a secondary container. Keep adding bicarbonate to your beaker until you see no \( \text{CO}_2 \) bubbles coming out. If you add it too fast, the content of your beaker will be spilled all over the place; this is why it is important to do this operation in a well-contained area. After you are done, you may want to use a 2-inch length of pH paper to check the pH of your waste solution. It should be about pH=8. If it is still acidic add a bit more bicarbonate. Once pH is around 8, you are allowed to dump the solution down the drain. Purge the drain with plenty of tap water.

For the remainder of the quarter, wash your glassware by rinsing extensively with DI water, followed by a final rinse with nanopure water. This is sufficient, since all the chemicals used in Chem 151L are water soluble and environmentally friendly. Do not use soap or detergent to wash your glassware at any time during the quarter. The lipids in soap coat the glass and change its hydrophilic properties. Treat the glass with the Nochromix mixture again if it becomes dirty. It is
to your advantage to keep this equipment very clean for the duration of the quarter. It is OK for it to be wet – volumetric glassware is designed to be used with water. Some analysts even prefer to keep unused glassware filled with distilled water to prevent surface contamination from dust and organics in the air.

IV. Calibration of Volumetric Glassware

Volumetric glassware is manufactured to specific standards, but it is not all identical and the manufacturing tolerances are not as strict as you may require for certain Chem 151L measurements. Indeed, small variations often occur from one piece of glassware to the next. It is possible to correct for systematic errors in the calibration markings, and such corrections are necessary for the most accurate analytical work. Section 2H of the textbook describes the calibration of volumetric glassware by weighting the amount of water the glassware holds with sensitive balances. The main goal of this section is to calibrate your 50 mL burette that you will using in all titrations.

All you have to do in this procedure is to weigh an empty, 150 mL conical flask; fill the burette with water up to the zero mark; transfer a certain amount of water into the flask; weigh the filled flask. The balances that read to ±0.001 g are sufficient for the burette calibration. **There is no need to weigh the burette in this procedure!**

The electronic balances in the lab have been calibrated recently by a technician, and you will not need to make a calibration. If you have evidence that a balance is not operating correctly, call this to the attention of your TA immediately.

You will need to know the density of water (Table 2-3 in the textbook) to convert the measured masses into volumes. The density of water is very sensitive to temperature, so a thermometer will be provided to measure water temperature. Record the temperature of the laboratory in your notebook, and use the corresponding density of water. You will not be required to make buoyancy corrections for weightings in this course.

See Table 6-2 provides an example of the data used to calibrate a 10 mL pipette. This table contains as many as 50 trials in order to get more reliable estimation of the measurement uncertainties. You will not have time to make so many measurements, but you should conduct a minimum of three trials to obtain a suitable calibration. Record your laboratory work in your laboratory notebook in proper format for this and all further experiments. In the lab notebook record at least three calibration trials for each volume of water drained from the burette.

Directions for using a burette are given in Section 2G-6; please follow them closely. When calibrating a burette it is very important to watch out for air bubbles in the spout. If bubbles are present in the spout, drain some water through the valve to get rid of them. Also note that poor burette reading technique will vitiate your calibration. Be sure to study Figure 2-21 that describes how to read the volume from a burette correctly. Most burette users record the position of the bottom of the meniscus, after adjusting the eye position to eliminate parallax.

**Do not get up on a tall stool to read a burette volume because it is unsafe. Use of a step stool is permitted.**

Calibration of the burette requires at least three trials at each of at least five volumes distributed evenly over the full range of the burette (15 measurements total). Use of a greater

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1. Check Table 2-3 carefully. What is the unit used? Is it density or the inverse of density?
2. This is the time (not later in the quarter) to learn the expectations and standards of your TA concerning the laboratory notebook entries.
number of volumes will result in a better calibration curve, if you have the time for it. The most effective way of calibrating the burette with the minimal number of weighings (18 times) is to do it in the following order:

- Weight an empty container (it does not need to be dry on the inside but it needs to be dry on the outside)
- Add 10 mL from the burette and re-weight the container with water in it
- Without disposing of water in the container, add 10 mL extra from the burette and re-weigh the container
- Keep going until you have reached 50 mL
- Get rid of water in the container and start over. Repeat the above procedure twice

The corrections for the burette should be graphed in your notebook as illustrated in Table 1 and Figure 2. Use Excel or a similar application to generate a similar graph for your data. In this graph the deviation between the volume delivered by the burette as measured by weighing the water delivered and the volume delivered as read from the burette scale is plotted. Once you have this calibration curve, you will be able to correct the volume read from the burette scale regardless of where on the scale you make a reading. You should use a full page in your notebook for this graph so later you can easily read out the corrections needed.

<table>
<thead>
<tr>
<th>Burette Calibration</th>
<th>Density of H2O (gm/mL):</th>
<th>0.997538</th>
</tr>
</thead>
<tbody>
<tr>
<td>Target volume (mL)</td>
<td>Initial weight of the contained (g)</td>
<td>1st run</td>
</tr>
<tr>
<td>10</td>
<td>101 151</td>
<td>101 325</td>
</tr>
<tr>
<td>20</td>
<td>111 103</td>
<td>111 342</td>
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<tr>
<td>50</td>
<td>141 296</td>
<td>141 333</td>
</tr>
</tbody>
</table>

Table 1: Sample calibration data for volume delivered by a burette

Figure 1: Sample correction plot for volume delivered by a burette. Standard deviation is used for the error bars in this example.
You will use your calibration information in several subsequent experiments. The correction can be applied in the following way:

**True delivered volume = Observed delivered volume + Correction**

To use the burette calibration graph for arbitrary volumes, you can assume that the correction is linear between each of the points at which calibration was actually carried out. For example, suppose you were to reach your titration end point at 25.00 mL. Reading from the above graph at 25 mL, we have an estimated correction of -0.062 mL (obtained by linear interpolation between 20 mL and 30 mL points). Therefore, the actual delivered volume is estimated to be 24.94 mL. Those of you who already took Chem 5 and studied MathCAD may want to program your correction into a MathCAD worksheet so that you can use it quickly. An example is given in Figure 2 below.

**Figure 2:** Sample MathCAD worksheet for calculating the corrected volume
Define the vectors containing the apparent volume and the correction (in mL)

VolumeVector := (0 10 20 30 40 50)ᵀ
CorrectionVector := (0 -0.0483 -0.0455 -0.0783 -0.0742 -0.0511)ᵀ

Construct a function to carry out linear interpolation between the data points

Correction(x) := interp(VolumeVector, CorrectionVector, x)

Now you can use it to correct your measured volume (all volumes are in mL)

Corrected1(x) := x + Correction(x)

Example: measured volume is x=25

Corrected1(25) = 24.938

An alternative way of doing this correction is by using a polynomial fit

\[
F(x) := \begin{bmatrix}
1 \\
x \\
x^2
\end{bmatrix}
\]

\[
d := \text{linfit}(\text{VolumeVector}, \text{CorrectionVector}, F)
\]

\[
\text{fit2nd}(x) := d \cdot F(x)
\]

Now you can use it to correct your measured volume (all volumes are in mL)

Corrected2(x) := x + \text{fit2nd}(x)

Corrected2(25) = 24.932

The burettes are too long to be stored in the drawers, so after cleaning and calibrating label your burette with a modest amount of tape so you can identify it again. Hand it to your TA to be stored in a common drawer specific to your lab section. Make sure your TA locks this drawer at the end of the lab section so that students from the following section do not take your burette by mistake.

Please be very careful handling the burettes for your own sake and that of your fellow lab partners. You will have put this effort into calibrating the burette, and if it breaks or chips you will need to start over.

V. Testing and Standardizing Your pH Meter

Once you are done with cleaning and calibrating your glassware, you should test your pH meter and standardize it with respect to buffer solutions. You will be using the meter in several; projects, and knowing how to standardize it quickly will save you a lot of time on these labs. You
will be using the same lab bench for every project, somewhere in the vicinity of your glassware drawer. pH meters have already been placed on the lab benches for your use.

Obtain 3 glass scintillation vials and fill them halfway with pH=4, pH=7, and pH=10 buffer solutions. These solutions should be on one of the lab benches; ask your TA if you cannot find them. Label your vials with the correct buffer pH. You will be using these buffer solutions for the rest of the quarter, so do not dispose of them when you are done. Use pH-meter calibration instructions available in the lab to test your pH-meter. If your pH-meter is not working, ask your TA for help with replacing the electrode.
VI. What to Submit in Your “Report”

1. At the end of this and all subsequent laboratory sessions you will submit to the TA the duplicate copies of all of the pages from your laboratory notebook, with proper dates, headings, data entries, hand-made graphs, and accompanying calculations, completed during the current laboratory session.

2. You should submit your versions of Tables 1 and Figure 1 prepared in Excel or comparable spreadsheet application. These should be submitted to your TAs before the second lab using EEE dropbox. Your figure should include errors equal to the standard deviations of your measurements, as shown in the above example. A copy of this figure must be printed and posted in your lab notebook for future use.

3. No formal report will be made on Project I. The points for this lab will be allocated as follows (note that it is very different from the remaining labs):

<table>
<thead>
<tr>
<th>Category</th>
<th>Points</th>
</tr>
</thead>
<tbody>
<tr>
<td>Correctness of prelab questions</td>
<td>20</td>
</tr>
<tr>
<td>Quality of lab note book entries</td>
<td>20</td>
</tr>
<tr>
<td>Cleanliness of your lab bench/hood at the end of the project</td>
<td>20</td>
</tr>
<tr>
<td>Correctness of your version of Table 1</td>
<td>20</td>
</tr>
<tr>
<td>Correctness of your version of Figure 1</td>
<td>20</td>
</tr>
</tbody>
</table>