Experiment 1: Multitechnique Operations

The purpose of this experiment is to provide practice with several of the quantitative methods you will use throughout this course. In later experiments your grade will depend on your mastery of these skills. For some experiments, a perfect score will require that your answers be well within 1% of the "true" composition of your "unknown" sample. On some experiments, an error of as much as 1 or 2% may result in scores as low as 40 out of 100 points. This experiment will not be graded, but success here is very important!

10 mL PIPETTE CALIBRATION

This is a simple measurement experiment to give you practice in pipetting, using the balance, and using your laboratory notebook. It may also give you a clearer idea of the calibration reliability of volumetric glassware.

This calibration is done by accurately weighing the volume of the liquid dispensed by the pipette. If the density of the liquid is accurately known at the temperature you are employing, the volume dispensed can be accurately determined. You will use deionized water.

Clean your 10 mL pipette so that no droplets of deionized water are left on the inside surface as it drains. Clean and dry a weighing bottle.

NOTE: For this experiment only, you may dry the weighing bottle using a Kimwipe. In future experiments it must be oven dried. If you have the time and want to gain experience, use the ovens to dry it to constant weight.

Weigh the bottle and its cap to the nearest tenth of a milligram. Use finger cots, gloves, or tongs to hold the bottle. Fingerprints often generate significant weighing errors!

Next, use a filling bulb to fill the pipette above the etched line with deionized water from a 250 mL beaker. Note the temperature of this water (Record room temperature using the large thermometer mounted on the wall near the balance room). MOUTH PIPETTING IS FORBIDDEN IN THIS COURSE! Dry the outside of the pipette with a Kimwipe and then release the finger pressure carefully to allow the liquid level to fall exactly to the etched line. The pipette should be held in a vertical position with the etched line at eye level. The bottom of the meniscus should coincide with the etched line. Touch the tip of the pipette to the side of the beaker to remove any drop formed at the tip. This manipulation is a bit tricky and may have to be repeated several times until you are sure you have the conditions you want. Once this is accomplished, touch the pipette to the inside of the weighing bottle, and allow the pipette to discharge its contents into the weighing bottle. It is best to allow the tip to touch the inner wall of the container near the bottom, but not in the liquid. Allow the pipette to drain itself for 20 or 30 seconds. DO NOT blow out the remaining portion of water in the tip! Remove the pipette. Cover the weighing bottle and weigh the bottle and its contents. This calibration should be done in triplicate and the three values should agree at least to within 1 part per thousand.

Common errors that occur in pipetting are a) warming of the pipette by holding the bulb portion in your hand, b) failure to allow sufficient drainage time, c) disturbing the residue that should remain in the tip, d) general carelessness in handling the weighing container and top, e) loss of water as you move from eye level to the weighing bottle (sudden movement will make some water squirt from the tip; slanting the pipette toward the horizontal before moving reduces this).
From the table of density vs. temperature for water (below), find the appropriate density and calculate the actual volume your pipette holds. Then answer the following questions.

1. What is the average amount held, from your three determinations? Calculate the standard deviation of your measurements.

2. What is the percent relative error in the 10.00 mL specification of the pipette? Is this within specifications listed on the pipette?

3. What was your precision in determining the volume of your pipette by this method? Express as relative standard deviation in parts per thousand.

### Relative density of water

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>0.9980</td>
</tr>
<tr>
<td>16</td>
<td>0.9979</td>
</tr>
<tr>
<td>17</td>
<td>0.9977</td>
</tr>
<tr>
<td>18</td>
<td>0.9975</td>
</tr>
<tr>
<td>19</td>
<td>0.9973</td>
</tr>
<tr>
<td>20</td>
<td>0.9971</td>
</tr>
<tr>
<td>21</td>
<td>0.9969</td>
</tr>
<tr>
<td>22</td>
<td>0.9967</td>
</tr>
<tr>
<td>23</td>
<td>0.9965</td>
</tr>
<tr>
<td>24</td>
<td>0.9962</td>
</tr>
<tr>
<td>25</td>
<td>0.9960</td>
</tr>
<tr>
<td>26</td>
<td>0.9957</td>
</tr>
<tr>
<td>27</td>
<td>0.9954</td>
</tr>
<tr>
<td>28</td>
<td>0.9952</td>
</tr>
<tr>
<td>29</td>
<td>0.9949</td>
</tr>
<tr>
<td>30</td>
<td>0.9946</td>
</tr>
</tbody>
</table>

### PREPARATION OF 0.1 M HCl AND QUANTITATIVE TRANSFER

In this experiment, you will prepare 500 mL of 0.1 M HCl.

**Procedure:**

1. Scrub a 250 mL beaker until no water droplets adhere when you rinse it.

2. Rinse the inside of the clean beaker with at least three small portions of deionized water. DO NOT DRY! This is the standard analytical technique for glassware preparation.

3. Measure about 100 mL of deionized water into the clean, rinsed beaker.
4. Clean a 10 mL graduated cylinder then rinse it with deionized water, as described above. Pour approximately 10-15 mL of reagent grade concentrated hydrochloric acid in a clean, dry beaker. Do not try to pour hydrochloric acid from the reagent bottle directly into the graduated cylinder. Next, rinse the graduated cylinder with 3 very small portions (< 1 mL) of reagent grade concentrated hydrochloric acid. Handle with care! Avoid breathing vapors. Discard the acid rinses into a 600 mL beaker half filled with tap water. Subsequently, the diluted waste may be washed carefully down the sink with copious amounts of tap water.

Note that it is a standard procedure to rinse volumetric glassware before use. Except for volumetric flasks, a second set of rinses (using the analytical solution) is generally also required.

5. Now measure 4 mL of concentrated HCl into the graduated cylinder.

6. Quantitatively transfer the HCl from the graduated cylinder into the water in the 250 mL beaker prepared earlier. (“Add Acid to water, like you oughter” and remember “A & W Root Beer”.)

7. Using your polyethylene wash bottle, liberally rinse the graduated cylinder with deionized water. Add rinses to the acid solution in the 250 mL beaker. After quantitatively and carefully transferring at least three such rinses, place about 3 mL of water in the graduated cylinder. Add one or two drops of 0.5 M AgNO₃. If the solution turns white or cloudy, your transfer was not quantitative. What is this white precipitate, and what is the reaction that produces it? If your transfer was incomplete, you may wish to repeat this whole procedure again. Discard the contents of the graduated cylinder down the drain.

8. Now, use a clean funnel to transfer the dilute acid solution from your 250 mL beaker into a clean 500 mL volumetric flask. Place a clean stirring rod across the top of the beaker, and use it to guide the liquid into the funnel as it is poured out of the beaker. This procedure is known as decanting.

9. Rinse the inside of the beaker, then the stirring rod, and then the funnel itself such that all rinse water goes into the volumetric flask. This is done with a lot of deionized water to insure that all the HCl is transferred to the volumetric flask.

10. Carefully rinse the tip of the funnel into your volumetric flask.

11. Now, to check your technique again, rinse the inside and outside of your funnel, and the stirring rod, into the beaker. Next rinse the walls of the beaker. Check your efficiency of transfer now by adding one or two drops of the 0.5 M AgNO₃ solution to the rinse solution. Again, if you get a white or cloudy result, your transfer was not quantitative.

12. Now take the quantitatively transferred solution that is in the 500 mL flask, and dilute this to volume with deionized water and mix thoroughly.

**USE OF THE BURETTE AND PRACTICE TITRATING**

Using the solution in your 500 mL flask, practice using your burette.

1. Clean the burette. Rinse with deionized water, then with small portions of your acid solution. BE SURE to rinse the tip of the burette by rotating the stopcock.
2. Fill the burette to a level below the 0.00 line.

3. Wait at least 30 seconds before taking the “zero” reading. Take zero reading and all burette readings using a burette reading card. Touch the card to the burette behind the graduations. The top of the black strip should be immediately below the meniscus.

4. Count 30 drops from the burette into an Erlenmeyer flask, and take the final reading. Then repeat this with 40 drops. Calculate the average volume of a drop for each trial. 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 and get half-drop endpoints. If in doubt, ask the instructor how to get half-drops.

5. Fill the plastic reagent bottle half full with deionized water. Add enough solid NaOH to make 1 L of 0.10-0.12 M NaOH and then fill to the shoulder with deionized water. Cap and mix thoroughly. You will standardize this solution later.

6. Use your 10 mL pipette to transfer 10.00 mL of your NaOH solution into a clean 250 mL Erlenmeyer flask. Rinse the sides of the flask with 20-30 mL of deionized water from your wash bottle.

7. Add 2-5 drops of phenolphthalein indicator (provided) to the Erlenmeyer flask. Note the color.

8. Titrate the NaOH solution to the phenolphthalein endpoint (pink $\rightarrow$ clear) using the HCl solution in your burette. Be sure to note the initial and final burette readings.

9. If time allows, repeat the titration using a fresh 10.00 mL portion of the NaOH. It should not be necessary to refill the burette. If your technique is good, the volume added should agree between replicates, to within better than 0.1 mL.

10. Save the unused base stock solution. (Be sure the NaOH is in the plastic container.) Rinse and clean all other glassware used. Store your burette and volumetric flasks filled with deionized water, if possible.

BONUS EXPERIMENTS (Due to your TA at the end of this laboratory period)

(10 bonus points) After completing the regular experiments, take a weigh bottle, and weigh it properly as before. Then wipe your bare hands on the bottle and weigh it again. Repeat this one more time. Report the values for proper weighing and improper weighing to a TA to receive credit.

(10 bonus points) After correctly completing the pipetting experiment, calibrate your 10 mL pipette incorrectly. Hold the bulb of the pipette in your hand for 2-3 min and then repeat the calibration. Next, use the filling bulb to blow out the remaining liquid in the pipette. Calculate the mass of water added for the two “incorrect” experiments along with the “correct” value and give them to a TA to receive credit.

(10 bonus points) Place two or three NaOH pellets in a weigh bottle and quickly weigh it. Place the open bottle on the bench for 5 minutes and then reweigh it. What did you observe, and what is your explanation for this observation?