Proper Handling of Analytical Equipment

Learning Objectives

- Introducing tools, techniques, and skills necessary for work in the analytical chemistry laboratory.
- Learning proper techniques for handling and making correct measurements with analytical equipment.
- Rinse analytical equipment precisely.

Additional Reading

Textbook: Quantitative Chemical Analysis, 9th edition, by Daniel C. Harris.

Materials

Analytical and top-load balances, 50-mL beakers, graduated cylinders, pipet, buret, 100-mL volumetric flasks, pennies, aluminum cylinder, spatula, tweezers, weigh boats, weighing paper, painters tape.

Reagents

$KMnO_4$

Background

The purpose of this experiment is to introduce several of the tools, techniques, and skills necessary for work in the analytical chemistry laboratory. The techniques should be considered one at a time, as unit operations. It is important to learn the proper techniques and to acquire individual skills before attempting additional laboratory experiments.

1.1. The Analytical & Top-load Balances

A balance is used to measure the mass of an object. It uses an electromagnet to balance the load on the pan. **Figure 1** shows a typical analytical balance used in the laboratory. Both the analytical and top-load balances are used for measuring small masses. In this experiment, you will obtain the mass of five new pennies - first, by determining the mass of each penny individually. Then you will determine the mass of all five pennies at once, remove one penny at a time, and calculate the individual masses of the pennies by finding the difference. The pair of masses determined for a particular penny by the two different methods should agree to within a few tenths of a milligram. From the data, you will determine the mean and median values, the standard deviation, and the relative standard deviation of the masses of the pennies. You will then weigh an unknown aluminum cylinder and report the mass of this unknown.

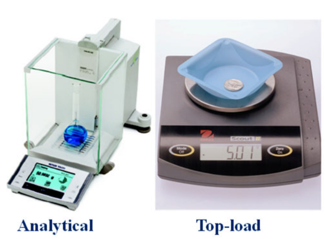

Figure 1: Analytical and top-load balances.

A standard mass in a balance is affected by buoyancy. Buoyancy is the upward force exerted on an object in a liquid or gaseous fluid. A buoyancy error occurs whenever the density of the object being weighed is not equal to the density of the standard mass.

The correct mass from a buoyancy error can be calculated as follows:

If m´ is read from a balance, the true mass m of the object in a vacuum is given by

$$m = \frac{m' \left(1 - \frac{d_a}{d_w}\right)}{\left(1 - \frac{d_a}{d}\right)}$$

where d_a is the density of air (0.0012 g/mL at 1 bar and 25 ºC), d_w is the density of the calibration weights (typically 8.0 g/mL), and d is the density of the object being weighed.

1.2. Burets

The buret is a precisely manufactured glass tube with graduations used for measuring the volume of a liquid delivered through a valve at the bottom. Because of surface tension between a liquid and glass, the surface of most liquids forms a concave **meniscus**. The quantitative chemist must learn how to accurately read the volume of a liquid forming a meniscus inside the buret. It is helpful to use black tape on a white card as a background for locating the precise position of the meniscus. A typical buret is shown in **Figure 2**.

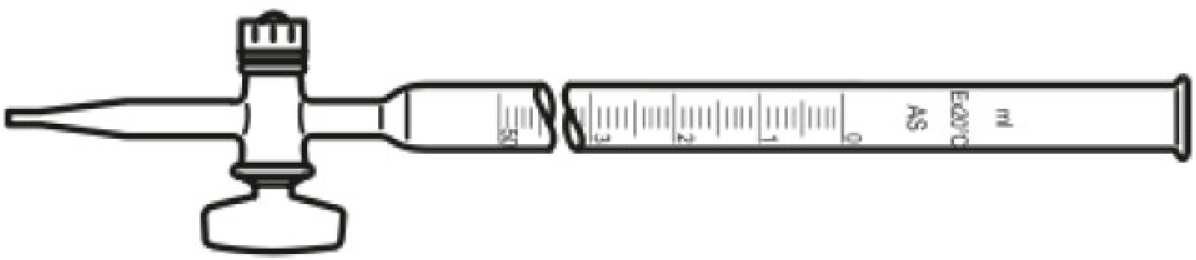

Figure 2: Buret.

1.3. Volumetric Flasks

The volumetric flask is one of the devices used to measure volume in the laboratory. A volumetric flask has been calibrated to contain a particular volume of solution at 20 ºC when the bottom of the meniscus is adjusted to the center of the mark on the neck of the flask. A volumetric flask is used to prepare a solution by first dissolving the desired mass of reagent in the flask with less than the final volume of liquid needed. The flask is swirled, and then more of the liquid is added and swirl the solution again. Adjust the final volume with as much well-mixed liquid as possible.

1.4. Pipets

Pipet delivers a known volume of liquid. The **transfer** pipet is calibrated to deliver one fixed volume. The last drop does not drain out of the pipet and should not be blown out. The **measuring** pipet is calibrated like a buret and will also deliver a variable volume. To deliver 5.4 mL, for example, you can start at 2.0 mL mark and terminating at the 7.4 mL mark. The transfer pipet is more accurate. **Figure 3** is an example of a typical volumetric flask and pipet.

Figure 3: Volumetric flask and a pipet.

Laboratory Procedure

Experiment A: The Analytical Balance

[1] After you have been instructed in the use of the balance, and have become familiar with its use, obtain a set of pennies, an unknown aluminum cylinder, and a pair of tweezers.

[2] Do not handle the pennies or the cylinder with your fingers; always use the tweezers. If you are using a mechanical balance, be sure to have the balance in the "off" or "complete arrest" position whenever removing anything from or adding anything to the balance pan.

[3] Before you begin to determine masses, zero your analytical balance carefully. Select five pennies at random from the beaker containing the pennies, and weigh each penny on your balance. Enter the data in your laboratory notebook. Keep track of the identity of each penny by placing each one on a labeled piece of paper.

[4] Check the zero setting on your balance. Place these same five pennies on the balance pan, determine their total mass, and record it.

[5] Remove one of the pennies from the balance, obtain the mass of the remaining four, and record the mass.

[6] Repeat this process, removing one penny at a time. Obtain the individual masses by subtraction. This process is known as weighing by difference, which is the way many mass determinations are done in the analytical laboratory.

[7] Repeat this process, but this time start from a single penny and increase the number of pennies on balance. Record the weight for each added penny until all five pennies have been added to the balance. Obtain the individual masses by subtraction.

[8] Finally, zero on your balance again, and find the mass of the unknown aluminum cylinder.

[9] In your report, construct a table or series of tables under the data and result section to report the data you have generated under this section correctly. Note that the data you created for **Lab 2** can be used in this lab.

[10] Calculate the true mass of the aluminum object.

Experiment B: Making Quantitative Transfers

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

Procedure

[1] Weigh a 50-mL beaker on an electronic top-load balance. Record the mass.

[2] Add about 0.0100 g solid $KMnO_4$ to the beaker and record the new mass. If you have a balance with a tare function, after weighing the empty beaker, depress the tare button to set the balance to zero. Then add $KMnO_4$ until the balance reads about 0.0100 g. Note that chemicals should never be returned to a stock bottle, as this may contaminate the bottle.

[3] Dissolve the potassium permanganate in the beaker using about 20 mL of distilled water. Stir 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 to a 100-mL volumetric flask fitted with a small funnel. To prevent solution from running down the outside of the beaker, pour it down the stirring rod, and then touch the rod to the spout of the beaker to remove the last drop. Add more water to the beaker, stir, and repeat the procedure.

[5] Repeat the procedure until no trace of the color of the permanganate remains in the beaker. Note the number of washings that are required to transfer the permanganate from the beaker to the flask quantitatively.

[6] Rinse the last portion of the solution from the stirring rod into the volumetric flask with a stream of water from the wash bottle. Rinse the funnel and remove it. Dilute the solution in the flask until the bottom of the meniscus is even with the graduation mark. Stopper, invert and shake the flask. Return it to the upright position, and allow the air bubble to return to the top of the neck.

[7] Repeat until the solution is entirely homogeneous; about ten inversions and shakings are required. Save the solution for the next part of the experiment.

[8] Summarize your result and present them in your report.

Experiment C: Delivering an Aliquot

Whenever a buret or pipet is used to deliver a measured volume of solution, the liquid it contains before measurement should have the same composition as the solution to be dispensed. The following operations are designed to teach the student to rinse and fill a pipet and also deliver an aliquot of the solution.

Procedure

[1] Fill a 10 mL pipet with the solution of potassium permanganate and let it drain.

[2] Draw a few milliliters of distilled water from a 50-mL beaker into the pipet, rinse all internal surfaces of the pipet, 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.

[3] Determine the minimum number of such rinse required to remove the permanganate color from the pipet altogether. If your technique is efficient, three rinses should be enough.

[4] Again fill the pipet with permanganate solution, and proceed as before. This time determine the minimum volume of rinse water required to remove the color by collecting the rinse in a graduated cylinder. Less than 5 mL is enough with efficient technique. In the rinsing operations, was the water in the 50-mL beaker contaminated with permanganate? If a pink color shows that it was, repeat the exercise with more care.

[5] As a test of your 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.

[6] Pipet 10 mL of the permanganate solution into a 250-mL volumetric flask.

[7] Carefully dilute the solution to volume, trying to mix the contents as little as possible.

[8] Mix the solution by repeatedly inverting and shaking the flask. Note the effort that is required to disperse the permanganate color uniformly throughout the solution.

[9] Rinse the pipet with the solution in the volumetric flask. Pipet a 10-mL aliquot of the solution into a 125-mL Erlenmeyer flask.

[10] Summarize your results and present them in your report.

Write a report based on the questions provided to you at the end of this handout.

Name: ________________________________

Partner's Name: ___________________________ **Date:** ___________

Pre-Laboratory Exercise

Show complete calculations were required to receive full credit. To be completed before the start of the lab. Note also that all pre-lab questions must be included at the end of your full lab report.

1. What do the labels "TD" and "TC" means for laboratory glassware? [**4 points**]

2. A sample of ferric oxide (Fe_2O_3, density = 5.24 g/mL) obtained from the ignition of a gravimetric precipitate weighed 0.4961 g in the atmosphere. What is the true mass in a vacuum? [**6 points**]

3. Describe how you will prepare 500.0 mL of 0.250 M K_2SO_4 with a volumetric flask. [**10 points**]

Suggested Tables

Table 1: Measurement of penny and aluminum mass determination. The pennies were measured using three different approaches. Each approach gives the mass of each penny.

<table>
<tr><td colspan="5">Individual Penny Measurement</td></tr>
<tr><td>Penny #</td><td>Weigh Boat (g)</td><td>Weigh Boat + penny (g)</td><td>Individual Penny Mass (g)</td><td>Average</td></tr>
<tr><td></td><td></td><td></td><td></td><td></td></tr>
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<tr><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td colspan="5">Penny Mass Measurement by Difference</td></tr>
<tr><td>Penny #</td><td>Total Mass (g)</td><td colspan="3">Individual Penny Mass (g)</td></tr>
<tr><td></td><td></td><td colspan="3"></td></tr>
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<tr><td></td><td></td><td colspan="3"></td></tr>
<tr><td></td><td></td><td colspan="3"></td></tr>
<tr><td colspan="5">Penny Mass Measurement by Summing</td></tr>
<tr><td>Penny #</td><td>Total Mass (g)</td><td colspan="3">Individual Penny Mass (g)</td></tr>
<tr><td></td><td></td><td colspan="3"></td></tr>
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<tr><td></td><td></td><td colspan="3"></td></tr>
<tr><td></td><td></td><td colspan="3"></td></tr>
<tr><td colspan="5">Aluminum cylinder measured mass</td></tr>
<tr><td colspan="2">Aluminum Cylinder Mass</td><td colspan="3"></td></tr>
</table>

Use this example to generate tables for experiment B & C.

Post-Laboratory Exercise

1. Provide a cover page for your report. The cover page must contain your **name**, **experiment title**, **course name**, and **date**.

2. Using the guide for writing reports posted in Canvas, write an abstract for all the data generated for the experiments in A, B, and C. The heading for this section should be entitled "**abstract**."

3. Using the guide for writing reports posted in Canvas, present all your data logically under a section entitled "**results**" in your report.

4. Using the guide for writing reports posted in Canvas, show your step-by-step calculations for the data generated for the experiments in **A**. The heading for this section should be entitled "**calculations**."

5. Using the guide for writing reports posted in Canvas, write a conclusion for all the data generated for the experiments in A, B, and C. The heading for this section should be entitled "**conclusion**."

6. Provide detailed answers to your pre-lab questions after your conclusion.

Due Date: **September 22, 2020**.

Submit your report via **<u>Turnitin in Canvas</u>** using the upload submission button. Hardcopy or email attachment reports are not acceptable.

Acceptable Report Format

The report must be submitted as a **Microsoft Word** document using the upload submission link. All identified articles must be submitted under the comment section of the submission link

All reports must contain a cover page with the following information: **Title of the experiment**, **student name**, **class (CHE 2121 - Quantitative Analysis Lab)**, **semester**, and **date**.
Font Style: **Times New Roman**

Font Size: **12-point**

Title Font Size: **14-point bold**

Student Name: **14-point bold**

Note: students who do not follow writing format instructions will be marked down.