

Experiment 2: Measuring Density and Calibrating Glassware CH2250: Techniques in Laboratory Chemistry, Plymouth State University

*NOTE: This lab consists of **TWO** parts (a and b). Be sure to do the pre-lab for both!*

Experiment 2a: Measuring Density of a Liquid and a Solid

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Suggested reading for background information: Section 2.3-2.6, *Exploring Chemical Analysis, 5th Edition*, Daniel C. Harris (2012).

Introduction:

Every measuring device has limitations on the number significant figures that can be read from it. In this lab you will measure mass and volume using instruments of differing precision and then calculate the densities of a liquid and a solid to the appropriate number of significant figures.

Density is a derived quantity (it cannot be measured directly, but is calculated through the measurement of more than one quantity) consisting of mass per unit of volume. In this lab, you will measure mass in grams and volume in milliliters (mL) and calculate density as g/mL. Mass will be measured with two types of balances, and volume will be measured directly using two types of graduated glassware.

NOTE: This lab includes steps on setting up tables and calculations. These steps will not be repeated in subsequent labs, but similar principles should be followed.

Purpose:

1. To learn the proper techniques for using an analytical balance and a three-arm balance.
2. To learn the proper techniques for using graduated cylinders and burets.
3. To perform calculations using the proper significant figures derived from your equipment.

Equipment:

- 100 mL beakers
- 100 mL graduated cylinder
- 50 mL buret
- buret-filling funnel
- 125-mL Erlenmeyer flask
- Rubber stopper for flask
- Sample of Unknown Liquid

Safety Considerations: Read through the procedures and note any safety considerations.

Procedure:

A. Prepare to Record Data

1. Make a table in your notebook containing the columns "Sample," "Initial Mass," "Final Mass," "Initial Volume," "Final Volume," and "Instruments." Be sure to label the table, starting with "Table 2a.1. Data to Calculate Density of a Liquid." (For the first table created in Experiment 2a). Include the proper units (mL, g) in the column headings.
2. Make entries in the table for the samples Liquid A1, Liquid A2, Liquid B1, and Liquid B2

B. Density of a liquid

1. Liquid A1: Place a rubber stopper on a 125 mL Erlenmeyer flask and record the mass in the appropriate place in your table using a three-arm balance. *After you record the mass DO NOT touch the flask or stopper with your hand. Use a Kim-wipe to hold these to prevent oils from your fingers changing the mass of the flask.*
2. Obtain a sample of Unknown Liquid from the instructor. You will use this Liquid for all the measurements in this section.



3. Fill a 100 mL graduated cylinder to at least 75 mL, but less than 100 mL with the Unknown Liquid. Record the volume. *Be sure to read the volume at the bottom of the meniscus with your eyes at the level of the meniscus and a sheet of paper held a few inches behind the cylinder.*
4. Pour about 5 mL of liquid from the 100mL graduated cylinder into the previously weighed Erlenmeyer flask. Replace the stopper on the flask immediately. Record the new volume. *Do not attempt to pour out "exactly" 5 mL--just get close and then record what you do.*
5. Using the three-arm balance, weigh the flask including the rubber stopper and the sample. Record the mass.
6. Dispose of the liquid in the sink. If the stopper has any liquid on it, dry it thoroughly. You do NOT need to completely dry the inside of the flask, but the outside should be dry.
7. Record the "Instruments" used (three-arm balance, 100 mL graduated cylinder).
8. Liquid A2: Replace the stopper on the flask. Using the three-arm balance, weigh the flask and record the mass.
9. Record the level of Unknown Liquid in the graduated cylinder. Repeat Steps 3 - 6 with about 30 mL of Unknown Liquid.
10. Liquid B1: Replace the stopper. Use an analytical balance to find and record the mass of the flask and stopper.
11. Fill a 50 mL buret above the top "0" line with the Unknown Liquid. Drain the liquid out of the buret to below the "0" line to dispel the air bubble from the tip. Record the initial volume. *Do NOT attempt to drain it "exactly" to the line! If the initial volume you record is "0.00" you will lose points on your report. Be sure to read the volume at the bottom of the meniscus with your eyes at the level of the meniscus and a sheet of paper held a few inches behind the buret.*
12. Dispense about 5 mL of Unknown Liquid into the flask. Immediately put the stopper back on the flask. Record the new volume of liquid in the buret.
13. Using the analytical balance, weigh the flask including the sample and the rubber stopper. Record the mass and dispose of the liquid in the sink.
14. Record the "Instruments" used (analytical balance, buret)
15. Liquid B2: Repeat Steps 11 and 13 with about 30 mL of Unknown liquid.

Data Analysis

A. Setting up Notebook for Analysis

1. Start the section "Data Analysis" in your notebook.

B. Density of Unknown Liquid

1. Make a table containing the columns "Sample," "Mass," "Volume," and "Density," Be sure to label the table, starting with "Table 2a.2" Make entries in the table for the samples Liquid A1, Liquid A2, Liquid B1, and Liquid B2.
2. Below the table, write the equation $\text{Mass} = \text{Final Mass} - \text{Initial Mass}$
3. Below that, find the mass of Liquid A1 using that equation. Label this as "Sample equation." Record this value in the table. *Be sure to use the proper number of significant figures.*
4. Find the masses of the other Liquid samples and record the value in the table. *You do not need to show these calculations in your notebook.*
5. Repeat steps 2-4 to find and record the volumes of the Liquid samples, using the equation:



$$\text{Volume} = \text{Final Volume} - \text{Initial Volume}$$

6. Find the density of the Liquid Samples in a similar manner with a "sample equation" using the equation: $\text{Density} = \text{Mass} \div \text{Volume}$

Conclusion

For the "Conclusions" portion of your lab, answer the following questions:

1. Which instruments resulted in the "better" determination (more significant figures) of the density of your liquid samples? Comment on the aspects of these pieces of equipment that made them better.
2. Was there a difference in the number of significant figures you found in the densities of the liquid when you used ~5 mL versus ~30 mL? Comment on why this may be the case.
3. To the least number of significant figures, were the densities of the liquid the same? If not, comment on which answer you trust more.

Homework Problems

See end of Experiment 2b for homework problems.



Experiment 2b: Calibration of Volumetric Glassware

CH2250: Techniques in Laboratory Chemistry, Plymouth State University

Adapted from "1. Calibration of Volumetric Glassware," *Experiments To Accompany Exploring Chemical Analysis, 4th Edition*, Daniel C. Harris, (2008), available at <http://www.whfreeman.com/exploringchem4e>.

Suggested reading for background information: Section 2.9, *Exploring Chemical Analysis, 5th Edition*, Daniel C. Harris (2013).

Introduction:

An important trait of a good analyst is the ability to extract the best possible data from his or her equipment. For this purpose, it may be desirable to calibrate one's own volumetric glassware (burets, pipets, flasks, etc.) to measure the volumes delivered or contained. This experiment also promotes technique in handling volumetric glassware.

NOTES:

- *This lab includes steps on setting up tables and calculations. These steps will not be repeated in subsequent labs, but similar principles should be followed.*
- *Start your Pre-Lab work for this part on the very next page in your lab notebook after where you started Experiment 2a. DO NOT leave blank pages between parts a and b that you will "fill in" after getting to lab.*

Equipment Needed:

- 50-mL Buret
- 10 mL volumetric transfer pipette
- pipette bulb
- 125-mL flask
- 100 mL beaker
- Rubber stopper for flask
- Thermometer

Safety Considerations: Read through the procedures and note any safety considerations.

Procedure:

A. Calibrating a 50-mL Buret

1. Create a table in the Results section of your notebook like the one below:

Table 2b.1. Data for Calibration of a 50 mL Buret.			
Temperature of Water:			
Mass of Flask + Stopper (g):			
Initial Volume (mL):			
Mass of Flask, Stopper, Sample (g)	Mass of Sample (g)	Final Volume (mL)	Total Volume Dispensed (mL)
<i>NOTE: include as many blank rows and space as you will need to record all your data</i>			

2. Fill a buret with distilled water that has been equilibrated to room temperature and force any air bubbles out the tip. See whether the buret drains without leaving drops on its walls. If drops are left, clean the buret with soap and water or soak it with cleaning solution. Adjust the meniscus to be between 0 and 1 mL, and touch the buret tip to a beaker to remove the suspended drop of water. Allow the buret to stand for 5 min before using it.



- Using an analytical balance, weigh a 125-mL flask fitted with a rubber stopper. *Hold the flask with a paper towel to prevent fingerprints from changing its mass.* Record the mass in the appropriate place in the table.
- If the level of the liquid in the buret has changed, tighten the stopcock and repeat the procedure. Record the level of the liquid as the Initial Volume in the appropriate part of the table.
- Drain approximately 10 mL of water at a rate < 20 mL/min into the weighed flask, and cap it tightly to prevent evaporation. Allow 30 s for the film of liquid on the walls to descend before reading the buret. Estimate volume to the nearest 0.01 mL. Weigh the flask, stopper, and sample. Record the volume (Final Volume) and mass in the table.
- Do NOT dump out the water! Continue collecting the water in the flask.
- Repeat step 5 three more times (until a total of about 40 mL has been dispensed from the buret).
- After the taking the final mass, measure and record the temperature of the water in the flask.

B. Calibrating a 10 mL Volumetric Pipette

- Create a table in the Results section of your notebook like the one below:

Table 2b.2. Data for Calibration of a 10 mL Pipette	
Temperature of Water: _____	
Mass of Flask + Stopper (g)	Mass of Flask + Stopper + Sample (g)
<i>NOTE: include as many blank rows and space as you will need to record all your data</i>	

- Fill a 100 mL beaker with distilled water that has been equilibrated to room temperature. Measure and record the temperature of the water.
- Weigh a 125-mL Erlenmeyer flask fitted with a rubber stopper. *Hold the flask with a paper towel to prevent fingerprints from changing its mass.* Record the mass in the appropriate place in the table.
- Fill a 10 mL volumetric pipette exactly to the line with the water. As you remove the pipette from the beaker, *gently touch the tip to the rim of the beaker to remove any drop of water.*
- Dispense the water into the 125 mL flask. Measure and record the mass of the sample and the flask in the appropriate place in the table.
- Dispose of the water in the sink. If the stopper has any liquid on it, dry it thoroughly. You do NOT need to completely dry the inside of the flask, but the outside should be dry.
- Repeat Steps 3-6 two more times.

Analysis

Calibration of 50 mL buret

- In Table 2b.1, calculate and fill in the columns "Mass of Sample" and "Total Volume."



2. Create a table like the one below in your Analysis section:

<u>Table 2b.3. Calibration of 50 mL Buret</u>		
Density Correction:		
Total Volume Dispensed (mL)	Calculated Volume (mL)	Correction (mL)
<i>NOTE: include as many blank rows and space as you will need to record all your data</i>		

3. Copy the "Total Volume Dispensed" data from Table 2b.1.
4. Look up the density correction factor for water at the temperature you measured in Table 2-5 (pg 54) in the textbook. Record this in the appropriate place in Table 2b.3, then use it to calculate and fill in the column "Calculated Volume" using the "Mass of Sample" data from Table 2b.1.
5. Calculate and fill-in the "Correction" column using the formula:

$$\text{Correction} = \text{Calculated Volume} - \text{Total Volume Dispensed}.$$
6. Prepare a calibration graph of Correction vs. Total Volume Dispensed in your notebook (your notebook should contain both horizontal and vertical lines allowing you to make the graph directly on the paper) like Figure 3-2 (pg 66) in the textbook.

Calibration of 10 mL buret

1. Create a table like the one below:

<u>Table 2b.4. Calibration of 10 mL Pipette</u>		
Density Correction:		
Mass of Sample (g)	Calculated Volume (mL)	Correction (mL)
<i>NOTE: include as many blank rows and space as you will need to record all your data</i>		

2. Calculate and fill-in the "Mass of Sample" data using the data from Table 2b.2.
3. Look up the density correction factor for water at the temperature you measured in Table 2-5 (pg 54) in the textbook. Record this in the appropriate place in Table 2b.4, then use it to calculate and fill in the column "Calculated Volume" using the "Mass of Sample" data.
4. Calculate and fill-in the "Correction" column using the formula:

$$\text{Correction} = \text{Calculated Volume} - 10.00$$
5. Calculate the average correction needed for your pipette.

Conclusions

1. Consider the correction factors you have calculated. Do you think this "error" is more a result of the equipment or your technique? What steps can you take to limit the errors from your technique?
2. Consider the size of the correction factor compared to the volumes that you may deliver by these pieces of glassware. In what cases is it important to consider the correction and when may it be ignored?

Homework Problems

The following problems from your book must be completed in your lab notebook (see the Syllabus for other suggested problems): Ch 2: **7, 10**; Ch 3: **1, 3, 4**

