MEASUREMENT HANDOUT
PART II

THE USE OF VOLUMETRIC WARE AND THE DETERMINATION OF DENSITY

In this experiment you will:

a) learn how to make volume measurements using a graduated cylinder, pipet, volumetric flask, and buret.
b) practice using the equipment listed above.
c) use the results of volume and mass measurements to determine the density of a liquid and a solid.

INTRODUCTION

The measurement of liquid volumes is an important part of many experiments. In some experiments volumes must be measured accurately, while in others less accuracy is required. The maximum accuracy possible is determined by the type of volumetric equipment used (Figure 3.1).

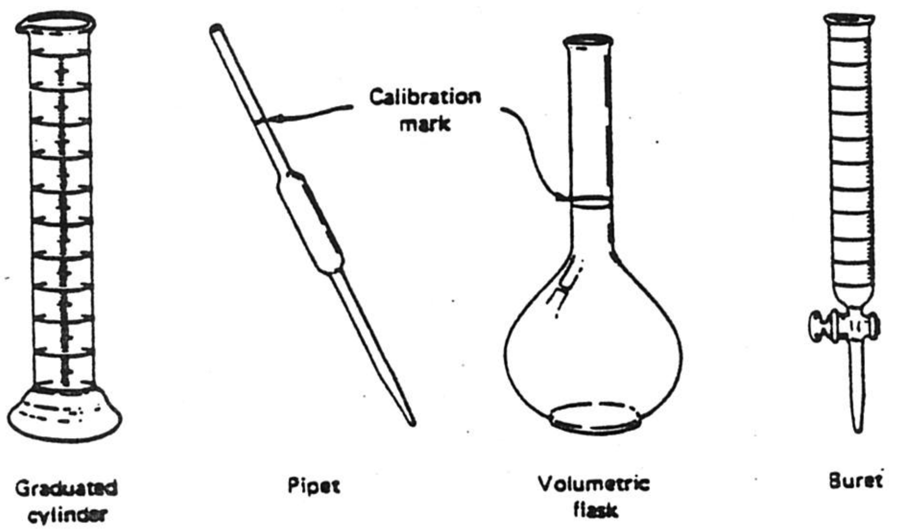


Figure 3.1. Commonly used volumetric glassware

EXPERIMENTAL PROCEDURE

A. The Graduated Cylinder

In order to properly use graduated cylinders and other volumetric ware, it is necessary to understand a little of the nature of the liquids involved. Water and most other liquids wet the surface of clean glass and, as a result, form a curved surface in glass containers. This curved surface, called a meniscus, becomes more apparent in narrow containers as shown in Figure 3.2. Volumetric readings are made at the bottom of the meniscus as represented in Figure 3.2c.

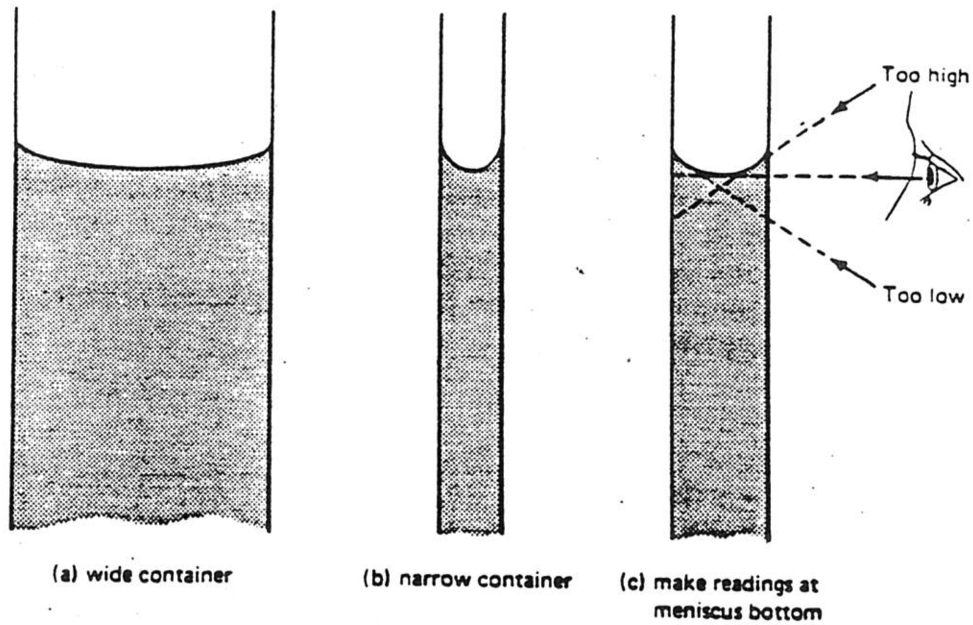


Figure 3.2. The appearance of a meniscus in volumetric glassware

Graduated cylinders are designed to measure any liquid volume up to the cylinder capacity. The volume contained in a graduated cylinder is estimated to one decimal place more than the smallest division on the cylinder. For example, a 50 mL cylinder has divisions corresponding to 1 mL. Volumes can be estimated to the nearest 0.1 mL by interpolating between the marks as shown in Figure 3.3.

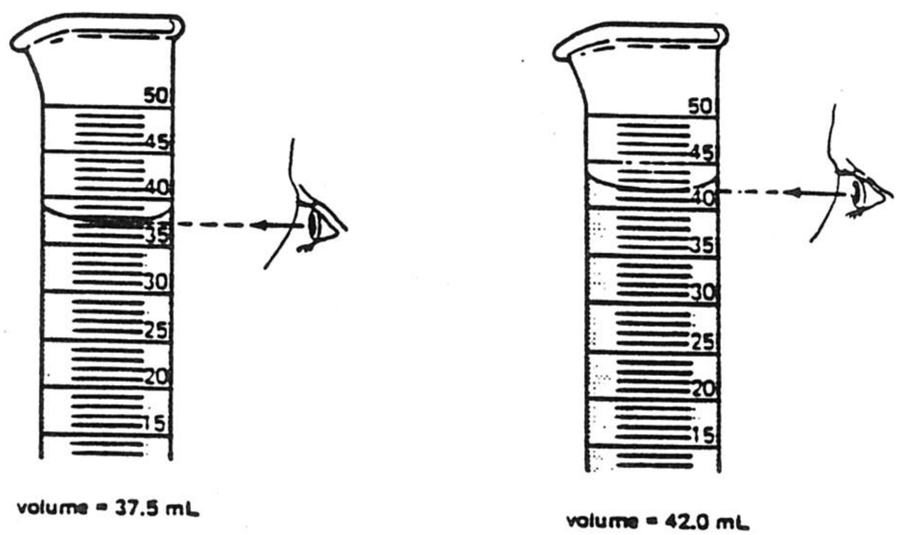


Figure 3.3. Volume measurements using a graduated cylinder

PROCEDURE

1. Obtain a 10 mL graduated cylinder from your desk equipment.
2. Weigh a clean, 50 or 100 mL beaker on a milligram balance and record the mass in Table 3.1 of the Data and Report sheet.
3. Use your wash bottle to add distilled water carefully to the graduated cylinder until, in your judgment, it contains exactly 10.00 mL. Remember to read the volume at the bottom of the meniscus. Record this volume in Table 3.1.
4. Pour the water sample from the cylinder into the weighed beaker.
5. Weigh the beaker and contained water on the same balance used in step 2, and record the mass in Table 3.1.
6. Without emptying the beaker, add a second carefully measured 10.00 mL sample of water from your graduated cylinder.
7. Weigh the beaker and contained water on the same centigram balance used before, and record the mass in Table 3.1.

B. The Pipet

Pipets are designed to deliver a specific volume of liquid and therefore have only a single calibration mark which is located on the narrow neck above the bulb (see Figure 3.1). *Pipets should never be filled by applying suction with your mouth.* A suction bulb should always be used. The proper procedure will be demonstrated by your instructor.

PROCEDURE

1. Obtain a 10 mL pipet from your desk equipment or the stockroom. Record the pipet volume in Table 3.2
2. Weigh a clean, dry 50 mL or 100 mL beaker on a milligram balance and record the mass in Table 3.2.
3. Use a pipet filler and draw distilled water from the beaker on instructor's bench into the pipet to a level above the calibration mark.
4. Bend over/down and, using the wheel, carefully adjust the water level until the bottom of the meniscus touches the mark.
5. Pressing the bar, allow the water to drain out all the way.
6. Touch the pipet tip to the side of the container of water to remove the attached drop. Or touch the surface of the water for a few seconds to completely drain. *Some liquid will remain in the pipet – it's supposed to.*

7. Weigh the beaker and water on the same milligram balance used in step 2. Record the combined mass in Table 3.2.
8. Without emptying the beaker, repeat steps 4-8. Record the mass of the beaker containing the two water samples in Table 3.2.

C. The Volumetric Flask

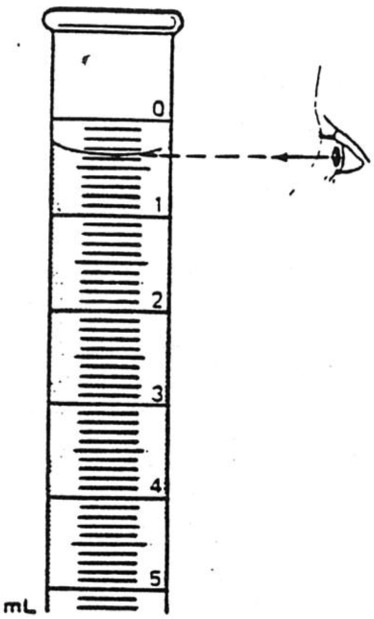
Volumetric flasks, like pipets, are designed to be used when a specific liquid volume is desired. Therefore, they also have only a single calibration mark which is located on the narrow neck of the flask (see Figure 3.1). However, unlike pipets, volumetric flasks are calibrated to contain a specific volume, not to deliver a specific volume when emptied.

PROCEDURE

1. Obtain a dry 100 mL volumetric flask from out front. Record the flask volume in Table 3.3.
2. Weigh the dry flask on the bench balance. Record the mass of the empty flask in Table 3.3.
3. Add H_2O from a beaker when nearly full . . . use your wash bottle to carefully add distilled water to the flask until the bottom of the meniscus just reaches the calibration mark.
4. Dry the outside of the filled flask and weigh it on the same balance used in step 2. Record the mass in Table 3.3.
5. Empty the flask and, without drying it, again fill it to the calibration mark with distilled water.
6. Dry the outside of the filled flask, weigh it on the same balance used in steps 2 and 4, and record the mass in Table 3.3.

D. The Buret

Burets are designed to deliver any precisely measured volume of liquid up to a maximum of the buret capacity. Once again, interpolation between scale divisions is necessary. Both 25 and 50 mL burets have scale divisions corresponding to 0.1 mL. Therefore, estimates between scale divisions will be written in terms of the next smaller decimal, 0.01 mL. Buret scales increase downward, so interpolations between divisions must also increase downward. Refer to Figure 3.4 for examples of buret readings, and ask your instructor for assistance as needed.



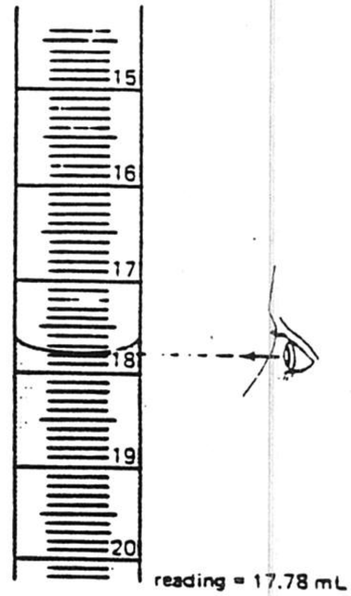


Figure 3.4. Examples of buret readings

PROCEDURE

1. Obtain a 50 mL buret from out front.
2. Mount the buret on a ringstand by means of a buret clamp as shown in Figure 3.5.
3. Fill the buret nearly to the top with distilled water.
4. Carefully open and close the stopcock a few times to acquaint yourself with its operation and also to remove any air bubbles from the tip of the buret. If any difficulties are encountered, ask your instructor for assistance.
5. When you are ready to proceed, refill the buret and adjust the level of the meniscus to be somewhere between 0.00 and 1.00 mL.
6. Read the water level to the nearest 0.01 mL, and record this initial reading in Table 3.4 in the blank corresponding to Sample 1.
7. Weigh a dry 50 mL or 100 mL beaker on a milligram balance and record the mass in Table 3.4.
8. Carefully add 10-12 mL of water from the buret into the beaker.

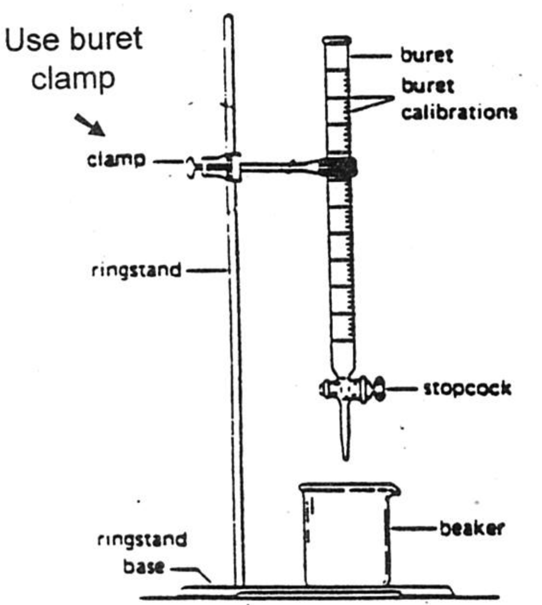


Figure 3.5. A buret mounted on a ringstand

9. Read the water level in the buret to the nearest 0.01 mL, and record this final reading in Table 3.4 in the blank corresponding to Sample 1.
10. Weigh the beaker and contained water on the same balance used in step 7, and record the mass in Table 3.4.
11. Do not refill the buret or empty the first water sample from the beaker.
12. Record the final buret reading from the first sample as the initial reading for the second sample in Table 3.4.
13. Add another 10-12 mL of water from the buret to the beaker.
14. Read the new water level in the buret to the nearest 0.01 mL and record it in Table 3.4 as the final reading for Sample 2.
15. Weigh the beaker and contained water on the same balance used before and record the mass in Table 3.4.

E. Density of an Unknown Liquid

In order to evaluate a density, both the volume *(v)* and mass *(m)* of a sample of substance must be determined experimentally. The density *(d)* is the ratio of these two experimental quantities:

$$d = \frac{m}{v} \qquad \textit{Eq 3.1}$$

PROCEDURE

1. Weigh a dry 50 or 100 mL beaker on a milligram balance and record the mass in Table 3.5.
2. Use the pipet to deliver a 10.00 mL sample of unknown liquid into the beaker. **Caution**: *Remember to use the pipet filler.* Record the pipet volume in Table 3.5.
3. Weigh the beaker and contents on the same balance used in step 3. Record the mass in Table 3.5.
4. Add another 10.00 mL of unknown to the beaker without emptying the first.
5. Weigh the beaker and contents on the same balance used in step 3, and record the mass in Table 3.5.

F. Density of a Solid

Solids can be weighed quite easily, but volume determinations are somewhat more difficult. If the solid has a regular geometric shape such as a cube or sphere, the volume can be calculated from the dimensions. You will work with an irregular solid, and determine the volume another way.

PROCEDURE

1. Obtain a solid #3 rubber stopper from your desk or the stockroom.
2. Weigh a dry 50 mL beaker on a milligram balance and record the mass in Table 3.6.
3. Place the rubber stopper in the weighed beaker, weigh them together, and record the combined mass in Table 3.6.
4. Fill 1 100 mL graduated cylinder approximately half way with distilled water.
5. Read the water level in the cylinder (remember to estimate to 0.1 mL), and record the value in Table 3.6 in the blank labeled "cylinder reading without stopped."
6. Carefully put the stopper into the cylinder without splashing out any water. This is best done by tilting the cylinder and letting the stopper slide down the inside wall.
7. Tap the cylinder so the stopper sinks to the bottom. Swirl the water gently to remove any clinging air bubbles from the stopper.
8. Read the water level in the cylinder that now contains the stopper. Record the level in Table 3.6.

CALCULATIONS AND REPORT

A. THE GRADUATED CYLINDER

With rearrangement, Eq. 3.1 can be written

$$v = \frac{m}{d}$$

Thus, the volume of a liquid sample is equal to the mass of the sample divided by the liquid density. We will use this relationship and the measured masses of water samples to calculate the volume of each sample. These calculated volumes will then be compared to the volumes obtained by reading the volumetric equipment. In each calculation, the density of water will be assumed to have a value of 1.00 g/mL. Thus, the volume calculated in milliliters will be numerically equal to the mass measured in grams.

EXAMPLE 3.1

Use the following data to calculate the volume of water contained in a graduated cylinder on a reading basis and a mass basis. Calculate the difference in volume between the mass and reading basis, and the percent difference.

Volume of water in cylinder	10.00	mL
Mass of beaker + sample	36.662	g
Mass of empty beaker	26.480	g

SOLUTION

The volume of water contained on a reading basis is simply the cylinder reading of 10.00 mL. The volume on a mass basis is calculated using the water mass, the water density of 1.00 g/mL, and Equation 3.2. The mass of water is obtained by subtracting the mass of the empty beaker from the mass of beaker plus sample: 36.662 g- 26.480 g = 10.182 g. The volume of this sample is obtained by using Equation 3.2 and the water density.

$$v = \frac{m}{d} = \frac{10.182\ g}{1.00\ g/mL} = 10.182\ mL$$

Thus, the water volume in milliliters is equal to the water mass in grams.

The difference in volume between the reading and mass bases is 10.182 mL - 10.00 mL = 0.18 mL.

The percent difference is calculated using Equation 3.3, given below in step 5.

$$\%\ difference = \frac{volume\ difference}{volume\ on\ a\ reading\ basis} \times 100 = \frac{0.18\ mL}{10.00\ mL} \times 100 = 1.8\%$$

---

1. Record in Table 3.7 the volume of water delivered according to the readings taken from the graduated cylinder.
2. Use Equation 3.2 and the water mass data in Table 3.1 to calculate the volume of water delivered (the volume on a mass basis). Note that the mass of the second water sample is obtained by subtracting the mass of the beaker containing the first sample from the mass of the beaker containing both samples. Record the calculated volumes in Table 3.7.
3. Determine the difference between the water volume on a mass basis and the volume on a reading basis for each sample. Record the difference in Table 3.7.

4. Calculate and record the average difference in volume by averaging the two values obtained in step 3.
5. Calculate the average percent difference between volumes on a mass and reading basis, using the following equation:

$$\text{Avg \% difference} = \frac{\text{avg. volume difference}}{\text{volume on a reading basis}} \times 100$$

6. Record the average percent difference in Table 3.7.

B. THE PIPET

Use the data in Table 3.2, and complete Table 3.8 as you did Table 3.7. Assume the pipet delivers exactly 10.00 mL when it is filled.

C. THE VOLUMETRIC FLASK

Use the data in Table 3.3, and complete Table 3.9. Assume the flask volume to be 100.00 mL when it is filled to the calibration mark.

D. THE BURET

Use the data in Table 3.4 and complete Table 3.10, using the following equation for *each sample*:

$$\text{\% difference} = \frac{\text{volume difference}}{\text{volume on a reading basis.}} \times 100$$

Note that the volume of water delivered according to buret readings is equal to the final reading minus the initial reading for each sample. The average % difference is obtained by averaging the two calculated % differences.

E. DENSITY OF AN UNKNOWN LIQUID

1. Record the unknown identification number in Table 3.11.
2. Record the volume of liquid (the pipet volume) used in each sample.
3. Use the data of Table 3.5 and determine the mass of each sample. Record the results in Table 3.11.
4. Use Equation 3.1 and the volume and mass obtained in steps 2 and 3, and calculate the density of each sample. Record these results and the average of the two values in Table 3.11.

F. DENSITY OF A SOLID

1. Use data from Table 3.6 and calculate the mass of the rubber stopper. Record the result in Table 3.12.
2. Use the two graduated cylinder readings of Table 3.6 to calculate the volume of water displaced by the stopper. Record this stopper volume.
3. Calculate and record the stopper density, using the mass and volume determined in steps 1 and 2, and Equation 3.1.

G. SUMMARY

1. Summarize the results of your volumetric measurements by completing Table 3.13. Record only the average % difference for the buret measurements.

DATA & REPORT SHEET

THE USE OF VOLUMETRIC WARE AND THE DETERMINATION OF DENSITY

fill to 10

DATA

A. The Graduated Cylinder

2 place

Table 3.1

Volume of water in cylinder	10.00 mL
Mass of beaker + both samples	69.796 g
Mass of beaker + first sample	60.219 g
Mass of empty beaker	50.706 g

B. The Pipet

Table 3.2

Pipet volume	10.00 mL
Mass of beaker + both samples	70.662 g
Mass of beaker + first sample	60.692g 60.676 g
Mass of empty beaker	50.706 g

C. The Volumetric Flask

Table 3.3

Volumetric flask volume	100.00 mL
Mass of flask + water (1st sample)	179.865 g
Mass of flask + water (2nd sample)	178.205 g
Mass of empty flask	79.803 g

Round answer to 2 places after the decimal

D. The Buret

100 mL Beaker 2 places

Table 3.4	Sample 1	Sample 2
Final buret reading	10.06 mL	9.00 mL
Initial buret reading	0.02 mL	9.02 mL
Mass of beaker + both samples	197.254 g 2nd 94.231 g	
Mass of beaker + first sample	1st 103.023 g	
Mass of empty beaker	50.706 g	

E. Density of an Unknown Liquid

Table 3.5

Unknown ident. number/letter	B
Pipet volume	10.00 mL
Mass of beaker + both samples	61.436 g
Mass of beaker + first sample	62.723 g
Mass of empty beaker	50.706 g

F. Density of a Solid

20 mL 1 place after decimal

Table 3.6

Mass of beaker + stopper	57.747 g
Mass of empty beaker	50.706 g
Cylinder reading with stopper	142.672 g
Cylinder reading without stopper	135.631

volume = mass / density   V = m / d

1.00 g/mL

DATA & REPORT SHEET

REPORT

A. The Graduated Cylinder

Table 3.7

	Sample 1	Sample 2
Water volume (reading basis)	10.00 mL	10.00 mL
Water volume (mass basis)		
Volume difference (*absolute* values)		
Average volume difference		
Average % difference		

[mass beaker + first sampl]
mass of water
subtract beaker w

[mass of beaker]

both samples -
2nd sample

neg - pos
pos - pos

B. The Pipet

Table 3.8

	Sample 1	Sample 2
Water volume (reading basis)		
Water Volume (mass basis)		
Volume difference (*absolute* values)		
Average volume difference		
Average % difference		

C. The Volumetric Flask

Table 3.9

	Sample 1	Sample 2
Water volume (reading basis)		
Water volume (mass basis)		
Volume difference (*absolute* values)		
Average volume difference		
Average % difference		

D. The Buret

Table 3.10

	Sample 1	Sample 2
Water volume (reading basis)	________	________
Water volume (mass basis)	________	________
Volume difference (*absolute* values)	________	________
% difference	________	________
Average % difference	________	

E. Density of an Unknown Liquid

Table 3.11

Unknown ident. number ________________

	Sample 1	Sample 2
Sample volume	________	________
Sample mass	________	________
Sample density	________	________
Average sample density	________	

F. Density of a Solid

Table 3.12

Stopper mass	____________
Stopper volume	____________
Stopper density	____________

G. Summary

Table 3.13

	Average Volume Difference	Average Percent Difference
Graduated cylinder	________	________
Pipet	________	________
Volumetric flask	________	________
Buret		________

QUESTIONS

1. Which of the following pieces of equipment is designed to deliver or contain a fixed volume of liquid?

   a) graduated cylinder b) pipet c) buret

   Explain your answer: ____________________

   ____

2. A 10 mL graduated cylinder has calibration marks representing each mL and 0.1 mL. Accurate readings should be estimated and recorded to the nearest

   a) 1.0 mL b) 0.1 mL c) 0.01 mL d) 0.001 mL

   Explain your answer: ____________________

   ____

3. Four students fill 100 mL volumetric flasks with distilled water and determine the mass of the water by a difference weighing. The water masses are given below as responses. In which case is it most likely that the student was reading the water level at the top rather than at the bottom of the meniscus?

   a) 99.8 g b) 100.1 g c) 100.0 g d) 100.2 g

   Explain your answer: ____________________

   ____

4. A student fills a buret with distilled water, adjusts the meniscus to a reading of 0.00 mL, and allows water to drain out until a reading of 10.00 mL is obtained. The water sample, weighed by difference, has a mass of 9.72 g. Which of the following experimental errors will account for the large difference between the volume according to readings and the volume according to mass?

   a) Initially the meniscus was above the 0.00 mL mark.
   b) The final meniscus was below the 10.00 mL mark.
   c) Air was cleared from the buret tip while draining the water, instead of beforehand.
   d) Water leaked from the buret into the beaker after the final buret reading was taken.

   Explain your answer: ____________________

   ____

5. A student determines the density of an unknown liquid by weighing a sample of known volume. The reported density is found by the instructor to be significantly higher than it should be. Which of the following experimental errors could account for this result?

a) The pipet used was unintentionally filled to a level above the calibration mark.
b) The balance was read incorrectly, and the recorded sample mass was lower than the true sample mass.
c) A part of the sample was spilled from the beaker before it was weighed.

Explain your answer: ____

____

6. A student determines the density of a metal object by first weighing it and then putting it into a graduated cylinder of water, just like Part F. The object sinks to the bottom of the cylinder. The student then calculates the density of the object and reports it as 0.36 g/mL. This result:

a) might be correct. b) is probably correct. c) cannot possibly be correct.

Explain your answer: ____

____