CHEM 121L
General Chemistry Laboratory
Revision 3.0

Density Determinations

- To learn about intensive physical properties.
- To learn how to measure the density of substances.
- To learn how to characterize a substance using its density.
- To learn different methods for measuring volume.

In this laboratory exercise, we will determine the densities of a Leaded Brass Alloy and a series of aqueous Table Sugar ($C_{12}H_{22}O_{11}$) solutions. The density of an aqueous Table Sugar solution of unknown composition will also be measured. This measurement can then be compared with the measured densities of the Table Sugar solutions of known composition to elucidate the Weight Percentage Table Sugar in the unknown sample.

Substances can be categorized according to whether they are heterogeneous, a solution, compound or elemental. Regardless of the substance's nature, it can be characterized by its various physical properties; mass, volume, color, density, etc. Physical properties of substances are themselves sub-categorized according to whether they are *intensive* or *extensive*. Extensive physical properties depend on the amount of substance being considered, whereas intensive physical properties do not. Some examples are:

Extensive	Intensive
Mass	Density
Volume	Color
Surface Area	Melting Point
	Boiling Point
	Vapor Pressure
	Surface Tension
	Conductivity

Intensive properties are particularly important because every pure substance has its own unique set of intensive properties which distinguish it from all other substances. And, even if the substance is not pure, its intensive physical properties can be useful in characterizing it. As an example, two metals which have a very similar appearance can be distinguished by considering their intensive physical properties:

Cadmium	
color:	silver white
melting pt:	320.9^{o}C
boiling pt:	765^{o}C
density:	8.642 g/cm^{3}
conductivity:	1.38x10^{5} cm$^{-1}\Omega^{-1}$

Chromium	
color:	steel gray
melting pt:	1890^{o}C
boiling pt:	2482^{o}C
density:	7.20 g/cm^{3}
conductivity:	7.74x10^{4} cm$^{-1}\Omega^{-1}$

A substance's density is a particularly important intensive physical property of that substance. The density of a substance, or object, is defined as the ratio of its mass to volume:

$$\text{density} = \frac{\text{mass}}{\text{volume}} \qquad \text{(Eq. 1)}$$

In effect, the density is a measure of the substance's compactness.

This property is important because it is extremely sensitive to the substance's environmental conditions and its composition. This is illustrated by looking at the variation in the density of Water with temperature and the variation in the density of Fuming Sulfuric Acid, Sulfuric Acid (H_2SO_4) infused with Sulfur Trioxide (SO_3), with composition:

Water

Temperature [^{o}C]	Density [g/mL]
0	0.99987
3.98*	1.00000
10	0.99973
20	0.99823
25*	0.99704
30	0.99567
40	0.99224
50	0.98807
60	0.98324
70	0.97781
80	0.97183
90	0.96534
100	0.95838

Fuming Sulfuric Acid

% H_2SO_4	Density [g/mL]
100	1.839
95	1.862
90	1.880
85	1.899
80	1.915
75	1.934
70	1.952

As can be noted from the above data, Water has a maximum density of 1.00000 g/mL at 3.98^{o}C, and the density decreases as the temperature rises. This is as is to be expected since as the temperature increases the added thermal energy causes the molecules comprising the sample to "bounce around" more, therefore requiring an increased volume, resulting in a lower density. It can also be noted, that unless we are performing high precision work, the density of water, near Room Temperature, can be taken as about 1.00 g/mL.

Because the density is a distinguishing characteristic of a given substance, it can be helpful in identifying it. Consider solutions of Table Sugar in Water. As the Table Sugar becomes more concentrated, the density of the solution increases. (This is the result of the tight interaction between the Sugar and Water molecules.) By knowing how the concentration affects the density, we can determine the Table Sugar concentration in a given sample by simply measuring its density. This basic procedure has several important industrial applications. Two common examples are the determination of electrolyte concentration in automobile batteries and the determination of antifreeze concentration in automobile cooling systems. In both cases, the density of each solution is measured with a hydrometer. The hydrometer is calibrated to give the concentration of the desired substance in terms of the solution density.

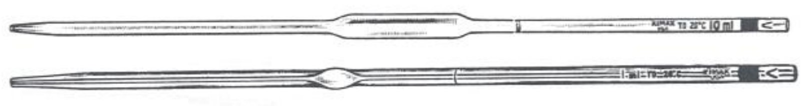
In a similar fashion, as the solution composition of Leaded Brass, primarily a mixture of Copper (Cu) and Zinc (Zn), with a little added Lead (Pb) to improve its machinability, is varied the density will likewise vary.

In this laboratory, we will first determine the density of Brass 306 by a direct measurement of its geometric parameters. We will compare this measured density with the accepted value in order to judge the accuracy of this technique. We will then establish the relationship between Table Sugar concentration in Water and solution density; i.e. we will establish a calibration curve relating the measured density and concentration. Finally, we will measure the density of a Table Sugar solution whose concentration is unknown. Use of the prepared calibration curve will allow us to establish the concentration of the Sugar solution.

In all cases, density measurements require an independent determination of the mass and volume for a sample of the substance under consideration. Mass measurements are quite easy. The pan balance was one of the earliest instruments employed by the medieval alchemists; current analytical balances easily measure mass to a tenth of a milligram (0.0001 g) precision and beyond. It is the measurement of volume which is difficult.

For liquids, several methods are available for measuring volume. Beakers and Erlenmeyer Flasks are frequently graduated with volume markings. However, these markings should be considered only approximate. Graduated Cylinders offer a significant improvement over Beakers and Flasks for volume measurements. (Think about why Graduated Cylinders are tall and narrow instead of short and fat. The design has specific implications for the precision of the device.) But once again, the precision of this device is still fairly low. For work requiring significant accuracy, a Volumetric Flask is required.

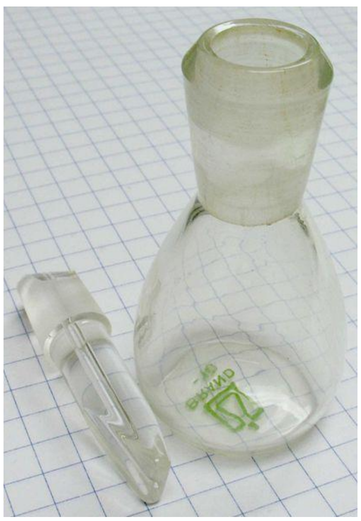
A pipette is a high precision volume measuring device used to transfer a given volume of liquid from one container to another. Volumetric Pipettes generally have a large bulb in the middle with a single mark along a narrow neck. By contrast, Measuring Pipettes are typically graduated much like a Graduated Cylinder.



(Volumetric Pipettes)

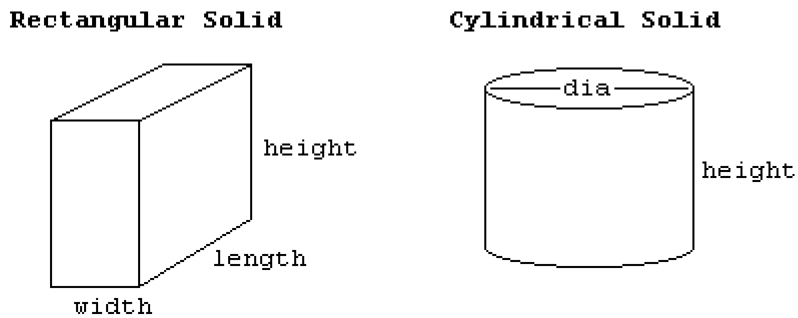
This is the device we will use to make our needed volume measurements of liquids.

For very high precision density measurements, a pycnometer (fr. Greek *puknos*, meaning density) is used to make the needed volume measurement. The pycnometer (one style is pictured below) is calibrated by determining the mass of Water required to fill it at a given temperature. The tabulated density of Water is then used to convert this mass to the volume of the device. The device can then be used to measure out a known volume of the liquid.



http://www2.volstate.edu/chem/1110/Density.htm

For regular solids, a simple ruled measuring device (ruler, caliper, etc.) can be used to determine the geometric parameters of the solid, and these parameters can be used to determine the volume:



For the above regular solids, the volume is related to the geometric parameters by:

Rectangular Solid
Volume = height x width x length

Cylinder
Volume = π (dia / 2)^2 x height

We will perform the following experiments:

1) Measure the density of a solid Brass cylinder by determining its volume by measuring its geometric parameters. This will then be compared to the accepted density to determine the accuracy of this procedure.

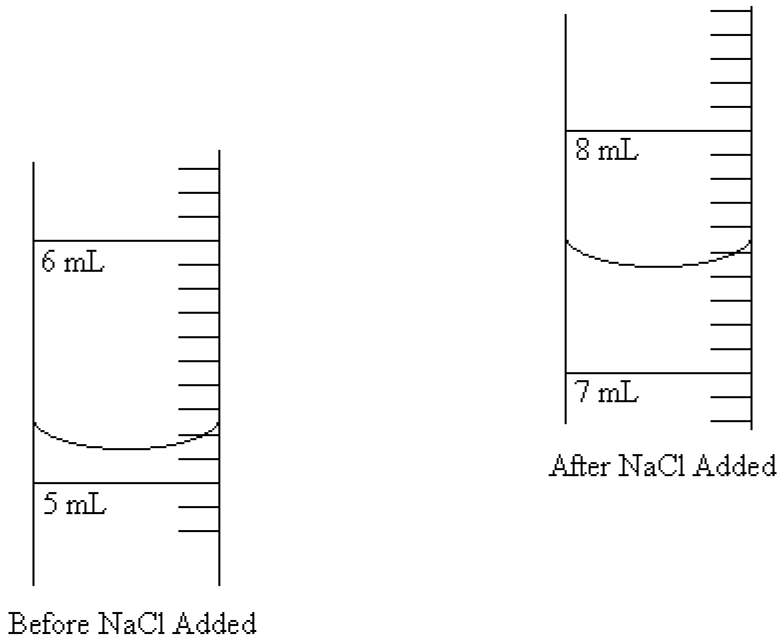
2) Prepare aqueous solutions of Table Sugar of known concentration. Measure each solution's density. Prepare a calibration curve for the density as a function of concentration.

3) Determine the density of a Table Sugar solution of unknown concentration. Use the calibration curve to determine the concentration.

In this experiment we will pay particular attention to the number of Significant Digits which can be recorded for each measurement. Therefore, you should review the material in the Appendix on “Measurement and Error”.

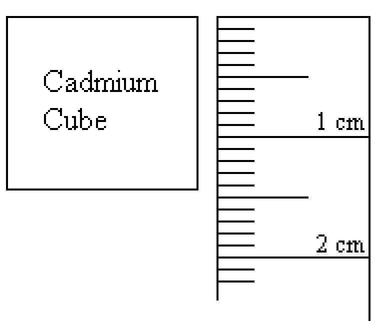
Pre-Lab Questions

1. The density of Table Salt (NaCl) is to be determined. It is found that Table Salt does not dissolve in liquid Hexane. Some liquid Hexane is added to a graduated cylinder, and the volume is read. Then, 5.03g of Table Salt is added to the graduated cylinder. The graduated cylinder is as pictured below:



   a) Report each of the above volume readings to the correct number of significant digits:
   b) Calculate the volume Table Salt used; to the correct number of significant digits.
   c) Calculate the density of Table Salt; to the correct number of significant digits:
   d) The value of the density of Table Salt, as reported in the *CRC Handbook of Chemistry and Physics*, is 2.165 g/mL. Calculate the percentage error for this measurement.

2. A 25.09g **cube** of Cadmium metal, an element which forms some of the most toxic salts known, has its volume measured with a ruler, as pictured below:



   a) What is the length of one side of the cube, reported to the correct number of significant digits?

b) What is the volume of Cadmium metal; reported to the correct number of significant digits?

c) What is the density of Cadmium; reported to the correct number of significant digits?

Procedure

Pay particular attention to the number of Significant Figures recorded for each of the following measurements. A significant portion of your grade will depend on the correct use of these Significant Figures.

Density of Brass 306

1. Obtain a cylinder of Brass 306 and measure its mass on the **Analytical Balance**. (If the cylinder is clean and dry, you may weigh it directly on the balance's pan. This is one of the few instances in which you can do this. Otherwise, always use weighing paper or a weighing boat to measure the mass of a sample.)

   !Good Practice – Always use the same balance for repeat weighings!

2. Measure the height of the brass cylinder using the digital calipers provided.

   Per the Instruction Manual
   1. **Lossen the lock knob.**
   2. **Push ON/OFF button to turn tool on.**
   3. **Check to ensure the caliper is completely closed by turning the thumb screw clockwise.**
   4. **Press the Zero button to confirm zero point.**
   5. **Press the inch/mm button to toggle between inches or millimeters.**
   6. **Rotate thumb screw in a counter-clockwise direction until the outside measure faces are slightly greater than the dimension of the object you wish to measure. Place the object in between the faces. Turn thumbwheel clockwise until faces contact the object.**
   7. **LCD display now shows dimension of object.**

3. Re-zero the calipers and repeat the measurement of the cylinder's height. Do this for a total of five measurements.

4. Report the average of these measurements, to the correct number of significant digits, in units of centimeters. (Be careful to correctly apply the definition of "significant digit" in this case!)

5. Repeat this procedure for the measurement of the cylinder's diameter.

Preparation & Density of Table Sugar Solutions

1. Each lab pair should work with a neighboring pair in preparing ~100mL of each of the following aqueous solutions:

5 wt% Table Sugar
10 wt% Table Sugar
15 wt% Table Sugar
20 wt% Table Sugar

All mass measurements required to prepare these solutions can be made using a **Top-Loading Balance**. The Sugar can be weighed directly in a 250 mL Beaker. The Water can be measured out in a 100 mL Graduated Cylinder.

For the 5% Solution:

i) Weigh out 5.00g of Table Sugar.
ii) Measure 95.0 mL of Water. (Assumes the density of Water is approximately 1.00 g/mL.)
iii) Add the Water directly to the Sugar in the beaker.

2. Stir the mixture until the Sugar is completely solvated. Complete solvation has been achieved when the solution is completely transparent. Any cloudiness suggests small particles of Sugar have not completely dissolved and more stirring of the solution is required.

3. Each lab pair should measure the density of each of the above solutions. This will be done by measuring a given volume of the solution from a pipette into a pre-weighed Erlenmeyer flask.

Practice Using the Pipette

4. First determine the temperature of the Room by measuring the temperature of a beaker of Water that has been allowed to equilibrate with the Room for several hours. This will be set-up for you in advance. (Note the arrangement used for the measurement.)

5. Now, obtain a clean, dry 25 mL Erlenmeyer flask with cork. Measure its mass using the **Analytical Balance**.

6. Use a 10 mL pipette to deliver deionized Water whose temperature has been allowed to equilibrate with the Room Temperature into the flask.

Correct use of the Pipette

1. **Use a pipette bulb to draw the liquid above the mark by evacuating the bulb, vertically inserting the pipette into the liquid and then drawing the liquid into the pipette.**
2. **Use a Kimwipe to dry to the pipette's stem.**
3. **Now place the tip of the pipette against the side of the container from which the liquid has been withdrawn and slowly drain the pipette until the liquid's meniscus is at the mark.**
4. **Move the pipette to the receiving container and allow the liquid to flow out (avoiding splashing) of the pipette freely. When most of the liquid**

has drained from the pipette, touch the tip to the wall of the container until the flow stops and for an additional count of 10. Most pipettes are To Deliver (TD) devices. In these cases, do not blow-out the last bit of liquid remaining in the pipette's tip.

7. Cork the flask and measure its mass.

8. Determine the volume of the pipette using the tabulated density of Water at the Room Temperature measured above. (Note: Be sure to correctly interpolate the table of densities provided in the Appendix.) **You need to do this calculation at this point.**

9. Use a Kimwipe to dry the flask as best as is possible. Re-cork and weigh the flask again.

10. Repeat steps 6 – 9 until three consecutive volumes have been delivered within the tolerance of the pipette. Average these volumes to determine the "calibrated" volume for the pipette.

11. Again measure the mass of the "dried" Erlenmeyer flask.

12. Beginning with your most dilute solution, use your pipette to deliver 10 mL of Table Sugar solution into the flask. **Before doing this "rinse" the pipette with some of the solution to be delivered. Simply draw some of the solution into the pipette and drain it into a waste container. You can repeat this if needed.**

13. Re-cork the flask and measure its mass.

14. Rinse the flask with some DI Water and dry it with a Kimwipe. Measure it mass.

15. Repeat these measurements for each Table Sugar solution, proceeding from the most dilute to the most concentrated. (Why do we proceed in this direction?)

16. Have your results approved by the lab instructor before moving on to the next measurement.

Density of Unknown Table Sugar Solution

1. Obtain an unknown aqueous Table Sugar solution. <u>Record the sample number.</u>

2. Measure the density of the sample as above.

Data Analysis

Each of the following calculated results must be reported to the correct number of Significant Figures. A significant portion of your grade will depend on the correct use of these Significant Figures.

Density of Brass 306 Cylinder

1. Determine the density of the brass cylinder based on the measured geometric parameters.

2. The reported density of Brass 306 is 8.47 g/mL. What is the percentage error for your density determination.

3. Comment on the quality of this method of density determination. Identify the single largest source of error in the determination.

Density of Table Sugar Solutions

1. Determine the density of aqueous Table Sugar at each of the Concentrations prepared. Use the pipette's "calibrated" volume determined in Step 10 of the procedure.)

2. Prepare a Calibration Curve of Density *vs.* Concentration (Wt %) for aqueous Table Sugar. This should be prepared using *Excel* or some other software package. Make sure the graph is labeled with an appropriate title and that each axis is also appropriately labeled.

   Be sure to include a 0% Sugar point; which will just be the density of pure Water. See the appendix below.

   Add an appropriate Trendline to this graph. Obtain the equation for the Trendline.

Density of Unknown Table Sugar Solution

1. Determine the density of your Unknown Table Sugar solution.

2. Use the above determined relationship between solution density and concentration (*i.e.*, the trendline) to determine the Weight Percentage Table Sugar in the Unknown solution.

Post Lab Questions

1. We wish to determine the density of a metallic alloy at 25^{o}C. This is done as follows. A pycnometer weighs 20.455g empty and 31.486g when filled with water at 25^{o}C. The density of water at 25^{o}C is 0.99704 g/mL. Pieces of the alloy are put into the empty, dry pycnometer. The mass of the alloy and the pycnometer is 28.695g. Water is added to the pycnometer to exactly fill it. The mass of the entire system is now 38.689g. What is the density of the alloy?

2. As noted in the Data Analysis above, our Brass sample has a reported density of 8.47 g/mL. What is the density of this sample in units of pounds/in^3? Why is the reporting of the density in units of pounds/in^3 problematic?

3. The *International Prototype Kilogram* is a right circular cylinder (height = diameter) machined out of a Platinum-Iridium alloy. If the Height of the prototype is 39.17 mm, what is the density of the alloy reported in units of g/cm^3?

Appendix - Tolerances for Class *A* Volumetric Pipettes at 20^{o}C

Capacity (mL)	Tolerances (mL)
0.5	0.006
1	0.006
2	0.006
5	0.01
10	0.02
20	0.03
25	0.03
50	0.05
100	0.08

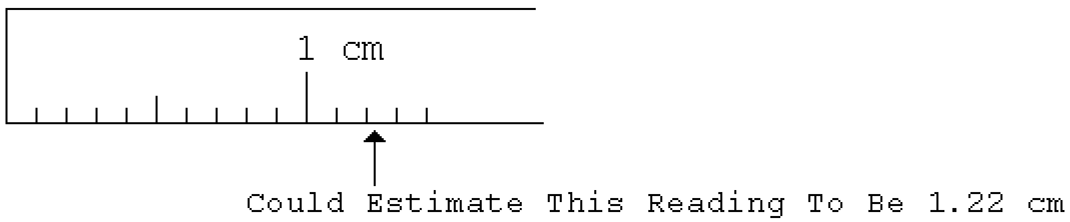
The Tolerances for Class *B* pipettes is typically twice that of a Class *A* device. (ASTM E694)

Appendix - The Density of Water

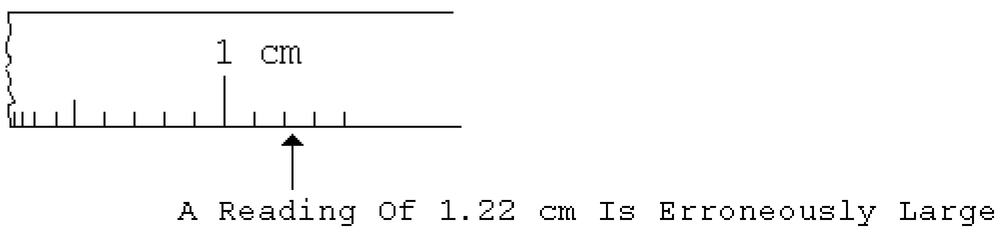
Temp [^{o}C]	Density [g/mL]
0	0.99984
5	0.99997
10	0.99970
11	0.99960
12	0.99950
13	0.99938
14	0.99925
15	0.99910
16	0.99895
18	0.99860
20	0.99821
22	0.99777
24	0.99730
26	0.99679
28	0.99624
30	0.99565
35	0.99403
40	0.99222
45	0.99022
50	0.98803
55	0.98570
60	0.98320
65	0.98056
70	0.97778
75	0.97485
80	0.97182
85	0.96862
90	0.96535
95	0.96190
100	0.95840

Appendix - Measurement and Error

We have to admit the possibility that our experiments are not perfect, and that some errors will infect the measurements made in the laboratory. Errors fall into one of two categories; Systematic or Random. Random errors result from imprecision in measuring devices; limited gradations, or rulings, and random fluctuations such as voltage irregularities in a resistance measuring ohmmeter. For instance, a ruler marked in 0.1 cm gradations can be used to measure a length with certainty to 0.1 cm. We can estimate lengths between the markings, but we will introduce a random error associated with that estimation.



Systematic errors result from improperly calibrated instruments or poorly designed experiments. An example would be a ruler that has been smashed at one end. The instrument, no matter how well gradated, will give an incorrect reading.



Systematic errors produce inaccurate results. Accuracy is how close our measurement is to the true result. One problem in detecting systematic errors is, we may not know the true result, and so will have no ability to know the error exists. Accuracy in a measurement can be represented by the Percentage Error:

$$\% \text{ Error} = |\text{True Value} - \text{Meas. Value}| \times 100 \, / \, (\text{True Value})$$

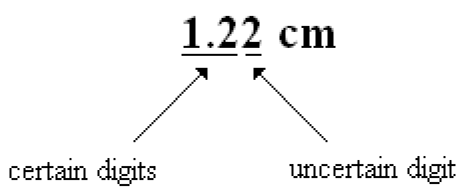
As an illustrative example, suppose we measure the density of a piece of solid Gold to be 19.47 g/cm^3. The accepted result for the density of Gold at 25^{o}C is 19.32 g/cm^3. Thus, the percentage error in our measurement is:

$$\% \text{ Error} = |19.32 - 19.47| \times 100 / 19.32 = 0.8\ \%$$

Note: The percentage error is typically reported to 1 or 2 significant digits. This is because this is a report of an error in a measurement, and not the measurement itself. Thus, a calculated percentage error of 3.25% would be reported as 3%. A calculation which results in a value such as 3.45% could be reported with 2 significant digits; 3.5%.

Random errors produce imprecise results. Precision is how repeatable our measurements are. If we make repeated measurements of a given quantity, the results can be averaged and the standard deviation calculated. The standard deviation is a measure of the random error, or imprecision, in the measurement.

Frequently we make only a single experimental measurement, instead of the desired multiple measurements. In these instances, the precision is represented by the number of Significant Digits in the measured result. These significant digits are those digits in a measurement which are certain, plus one which is uncertain. Frequently, the uncertain digit is due to estimating the position between the gradations on the measuring instrument, as in the case of the ruler measurement above.



As always, repeated work gives us a better estimate of the random error in our measurement, but the significant figure approximation works surprisingly well.