Microscale Density And Refractometry

INTRODUCTION

In this experiment, the density of an unknown liquid will be determined using microscale techniques. By using the density, the student should able to identify their liquid from a table of compounds.

DISCUSSION

Density is an example of an intensive property of matter. That is, the density of a substance does not depend on the amount of the substance. The water in a single drop of water has the same density as the water in a 5-gallon pail of water, assuming they have the same temperature. Since the density of a substance can easily be obtained, even with a very small sample, it is often used to aid in the identification of an unknown substance. In some cases, it may also aid in determining the composition of a mixture of known substances. For example, when the liquid in an automobile radiator is tested to check its freezing temperature, the specific gravity (closely related to the density) of the liquid is measured and the freezing point is determined from a table. This is possible because it is assumed that a radiator contains a mixture of water and antifreeze. Different mixtures of water and antifreeze yield different densities. The density of a substance is defined as its mass per unit of volume, calculated as follows:

$d = \frac{m}{V}$, where d = density, m = mass, and V = volume.

The refractive index of a substance is another unique physical property that can aid in the determination of the identity of an unknown substance. The index of refraction can be measured with great accuracy (up to 8 decimal places) and consequently can be very useful in characterizing pure organic liquids. As with density, refractive index can also be used to assess the purity of known liquids and to determine the percent composition of known solutions. When a beam of light passes through a liquid, its velocity is reduced, causing it to bend downward. The extent that the light is bent when it passes through a liquid depends upon the identity of the liquid. The angle at which the light is bent is the characteristic that is measured in a refractive index determination.

PROCEDURE

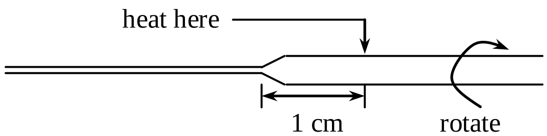
CAUTION: Be careful when handling a Pasteur pipet. It has a sharp end that may cause cuts or puncture wounds if handled carelessly. You will also be heating glass over a Bunsen burner. Since hot glass and cold glass look alike, use caution to avoid burns. If you receive a cut or burn, notify the instructor immediately, even if it is minor.

DISPOSAL: Dispose of all glass in one of the designated sharps containers in the laboratory. Liquid unknowns should be disposed into a designated waste container under one of the hoods in the laboratory.

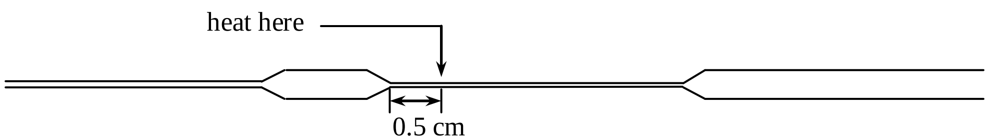
REMINDER: Use your laboratory notebook to record all data. Entries must be made in pen.

Part 1: Making a micropycnometer

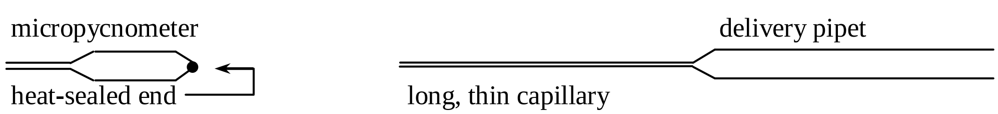
1. Obtain a Pasteur pipet. See caution above. Connect a Bunsen burner to the gas and ignite the burner. Adjust the flame to obtain a hot blue flame. Place the Pasteur pipet in the hottest part of the flame, about 1 cm from the capillary stem. Rotate the pipet in the flame to achieve even heating. When the glass becomes very soft, quickly remove the pipet from the flame and pull the two ends apart rapidly to yield a long thin capillary. Hold the pipet stretched and steady until the capillary hardens.



2. To separate the micropycnometer, heat the capillary about 0.5 cm from the end. Continue heating the pycnometer to seal and yield a smooth end.



3. If necessary, cut the open end of the pycnometer to obtain a shorter neck and fire polish the opening. Use caution to avoid sealing the open end. Also, the opening of the pycnometer must be large enough for the pipet capillary to fit inside, as well as to allow air to be displaced from inside. If the neck of the pycnometer is too long, there is a greater chance of having the capillary break off inside.



4. If the long, thin capillary of the delivery pipet fits easily into the pycnometer, attach a rubber bulb to the delivery pipet and move on to Part 2. If it does not easily fit, construct a new micropycnometer and/or delivery pipet.

Part 2: Finding the volume of the micropycnometer

> **NOTE:** Since you will be calculating differences in mass that are very small, it is important to avoid introducing anything to the beaker or stopper that might cause the mass to change. This includes drops of water, tiny fragments of glass or dirt, or even fingerprints. From this point forward, the beaker, stopper, and micropycnometer should be handled only with KimWipes® and/or gloves.

5. Obtain a one-hole rubber stopper. The hole in the stopper should be large enough for the pycnometer to sit without tipping.

6. Wipe the pycnometer clean and place it into the hole of the stopper. Place the entire assembly onto an analytical balance and record the mass.

7. Place some water into a small beaker or vial. Measure and record the temperature of the water using a thermometer.

8. Draw some water into the delivery pipet. ***Tip:*** *Avoid spilling any liquid into the beaker or onto the stopper. You may use a second one-hole stopper as a pycnometer stand.* Insert the tip of the pipet into the pycnometer and fill it with water. As the pycnometer is filled, slowly withdraw the capillary tip to avoid getting air bubbles trapped inside the pycnometer. Thoroughly wipe and dry any water that may have spilled over the pycnometer.

9. Place the pycnometer back into the hole of the stopper and measure the mass. Calculate the mass of water in the pycnometer. Withdraw the water from the pycnometer using the delivery pipet.

10. Repeat steps 8 and 9 to yield a total of four trials.

11. Calculate the volume of the micropycnometer for each trial using the mass and density of water from the table below. Determine the average volume of the pycnometer.

Density of Water			
Temp (°C)	Density (g/cm^3)	Temp (°C)	Density (g/cm^3)
18.0	0.99860	22.0	0.99777
18.5	0.99850	22.5	0.99766
19.0	0.99840	23.0	0.99754
19.5	0.99830	23.5	0.99742
20.0	0.99820	24.0	0.99730
20.5	0.99810	24.5	0.99717
21.0	0.99799	25.0	0.99704
21.5	0.99788	25.5	0.99691

Part 3: Determining the Density of an Unknown Liquid

12. Obtain a vial that contains an unknown liquid. Record the vial number.

13. Before making any measurements, the micropycnometer must be washed with acetone followed by a wash with some of your unknown liquid. To do this, draw some acetone into the delivery pipet. Insert the tip of the pipet into the pycnometer and fill it with acetone. Next, withdraw the acetone from the pycnometer using the pipet. Repeat the filling and withdrawing two more times to ensure that all of the water has been removed. Repeat this wash procedure three times with your **unknown liquid** to now ensure that you have removed all of the acetone from the pycnometer.

14. Fill the pycnometer with your unknown liquid and avoid trapping any air bubbles inside. Thoroughly wipe and dry any liquid that may have spilled over the pycnometer.

15. Place the pycnometer into the stopper and beaker assembly and measure the mass. Calculate the mass of liquid in the pycnometer. As with the water withdraw and refill the pycnometer to yield four trials.

16. Calculate the density of the unknown liquid for each trial using the mass of the liquid and the average volume of the pycnometer. Determine the average density of the liquid.

17. Determine the refractive index of your unknown liquid. (Your instructor will demonstrate how to use a refractometer). Wipe off the unknown liquid with a KimWipe® and repeat the determination of your refractive index one more time with a new sample of your unknown liquid. When you are finished, wash the prism with a SMALL amount of acetone and dry it again ***gently*** with a KimWipe®.

18. Use the table below to identify your unknown.

Substance	d_4^{20} (g/mL)	n_4^{20}	$b.p._{760}$ (°C)
Cyclohexane	0.7786	1.4266	80.7
Cyclohexanol	0.9624	1.4641	161.1
Diethyl ether	0.7134	1.3550	34.6
Ethyl acetate	0.9003	1.3723	77.1
Hexane	0.6603	1.3751	69.0
Isopropyl alcohol	0.7851	1.3772	82.5
Methyl alcohol	0.7915	1.3292	64.7
Toluene	0.8669	1.4961	110.6

PRECISION OF DATA

At this stage, you have probably determined the identity of your unknown. However, you have not determined the precision of your data. The precision of your data may be determined by using a statistical calculation called the standard deviation. To learn more about calculating various statistical functions such as the mean and standard deviation, go to the Statistics link at: http://webs.anokaramsey.edu/chemistry, click on “Chem 1061”, then click “Microscale Density”, then “Chemistry Statistics”.

For help with various calculators, go to one of the following links. In particular, the Texas Instruments link contains a wealth of information, including online manuals.
Texas Instruments: http://education.ti.com/
Casio: http://www.casio.com, follow the links to “Calculators”
Sharp: http://www.sharp-usa.com/products/FunctionLanding/0,1050,4,00.html
Hewlett-Packard: http://www.hp.com/calculators

LABORATORY REPORT

Include the following information in your laboratory report, due at the beginning of the laboratory period next week. *At your instructor’s discretion, individual or pair reports may be required.*

1. Title (including name, partner’s name, etc.)

2. A description of the procedure you followed to produce the experimental data and results. Alternatively, you may reference the procedure by citing the URL’s (web addresses).

3. Results. A table or tables of your experimental data, clearly labeled with the proper units. Remember to use subscripts and superscripts where appropriate (examples: cm^3 or H_2O). For each type of calculation performed, give the formula used to perform the calculation, rearranged to yield the desired variable. Alternatively, you may give one sample calculation for each type of calculation performed. These formulas and calculations may be neatly handwritten or typed. You may also use Microsoft *Equation 3.0* to input equations. A tutorial for *Equation 3.0* may be found at http://webs.anokaramsey.edu/chemistry/Chem1062/Labs/WritingTools/WritingTools.doc.

   Calculate the standard deviations for volume of the pycnometer and for the density of the unknown. Use the standard deviation to determine the proper number of significant figures in the mean volume of the pycnometer and the mean density of the unknown (round these values to the correct number of significant figures).

4. Discussion. As part of the discussion, be sure to identify your unknown liquid and provide the evidence for your claim. You should also address any uncertainty in either the experiment or your calculations and how it affected your results. Other topics which may be included are things like whether or not the purpose of the lab was met and new concepts learned.

5. References, properly cited, to any sources you may have used to perform, complete, or analyze the results of the lab.

6. For the complete guidelines for preparing individual laboratory reports, go to http://webs.anokaramsey.edu/chemistry. Click on "Chem 1061", then click on "Laboratory Reports". The reports will be due at the beginning of the lab period for your next regularly scheduled lab.

7. **Follow your instructor's directions for submission of the lab report.** You may have the option of submitting this lab by email, through the D2L dropbox, or on paper. If you choose to submit by email, please attach a single file with a filename convention of *Lastname Firstname Microscale* and a subject line of "Chem 1061: Microscale Density".

Ideas for major portions of this lab were obtained at the 150th $2YC_3$ Conference: Microscale Chemistry in the 21st Century, Fort Smith, AR, November 5-6, 1999.