Experiment 2
Microscale Density and Refractometry

OUTCOMES

After completing this experiment, the student should be able to:

- make a micropycnometer from a Pasteur pipet.
- calculate the density of a liquid using a pycnometer.
- determine the refractive index of a liquid using a refractometer.
- identify an unknown liquid based on its density and refractive index.

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 five gallon pail of water, assuming they have the same temperature. Since the density of a substance can be easily 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:

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

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

⚠ ***Wear safety glasses or goggles at all times for this experiment.***

⚠ ***Be careful when handling the Pasteur pipet. It has a sharp end that may cause cuts or puncture wounds if handled carelessly.***

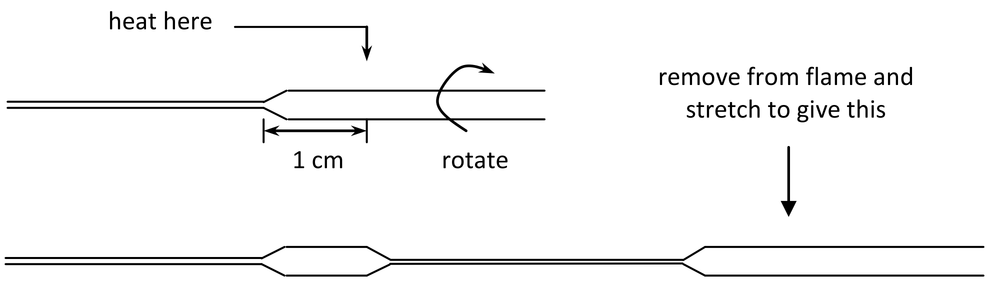
⚠ ***Use the Bunsen burner with care. Do not leave the burner unattended.***

⚠ ***Hot glass and cold glass look the same. Use caution to avoid burns.***

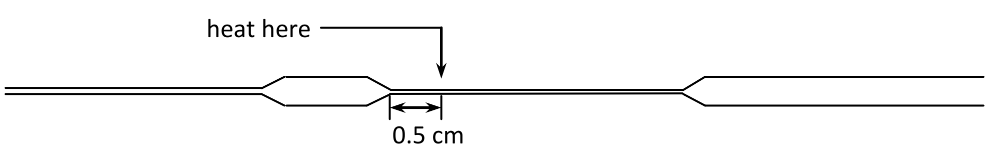
⚠ ***Never pipet by mouth.***

Procedure 1: Making a Micropycnometer

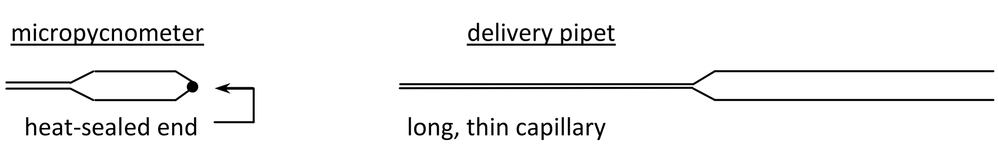
1. Obtain a Pasteur pipet. Connect a Bunsen burner to the gas and ignite the burner. Adjust the flame to obtain a hot blue flame. Place the Pasteur pipet in the hottest part of the flame, about 1 cm from the capillary stem. Rotate the pipet in the flame to achieve even heating. When the glass becomes 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.



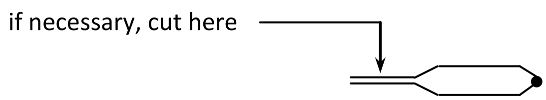
2. To separate the micropycnometer, heat the capillary about 0.5 cm from the end. Continue heating the pycnometer to seal and yield a smooth end.



to produce the following two objects:



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 necessary, cut the end of the delivery pipet to make sure that it is not sealed. 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 Procedure 2. If it does not fit easily, construct a new micropycnometer and/or delivery pipet.

Procedure 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 into the micropycnometer assembly 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, all glass should be handled only with KimWipes® and/or gloves.

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

2. Obtain two one-hole stoppers. The holes in the stoppers should be large enough for the pycnometer to sit without tipping. Use a piece of tape to label one of the stoppers "balance" and the other stopper "transfer."

3. Wipe the pycnometer clean and place it into the hole of the "balance" stopper. Place the entire assembly onto an analytical balance (with doors) and record the mass.

4. Place the pycnometer into the hole of the "transfer" stopper. Draw some water into the delivery pipet. Insert the tip on 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.

5. Place the pycnometer into the hole of the "balance" stopper and measure and record the mass of the entire assembly on an analytical balance.

6. Place the pycnometer back in the hole of the “transfer” stopper and withdraw the water from the pycnometer using the delivery pipet.

7. Repeat steps 4 to 6 three more times to yield a total of four trials.

Procedure 3: Determining the Density of an Unknown Liquid

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

2. Before making any measurements with the unknown liquid, all water must be removed from the micropycnometer and delivery pipet. Place the pycnometer into the hole of the “transfer” stopper. Draw some acetone into the delivery pipet, insert it 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 with acetone to ensure that all of the water has been removed. Repeat this was procedure three times with your **unknown liquid** to no ensure that you have removed all of the acetone from your pycnometer.

3. With the pycnometer in the “transfer” stopper, 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.

4. Place the pycnometer into the hole of the “balance” stopper and measure and record the mass of the entire assembly on an analytical balance.

5. Place the pycnometer back in the hole of the “transfer” stopper and withdraw the unknown liquid from the pycnometer using the delivery pipet.

6. Repeat steps 3 to 5 three more times to yield a total of four trials.

Procedure 4: Determining the Refractive Index of an Unknown Liquid

NOTE: This procedure may be done at any time during the lab.

1. Determine the refractive index of your unknown liquid. (Your instructor will demonstrate how to use a refractometer.) Note that there are no units for the refractive index.

2. 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.

3. When you are finished, wash the prism with a SMALL amount of acetone and dry it again **gently** with a KimWipe®.

⚠ ***Dispose of all glass in the broken glass container.***
⚠ ***Dispose of all chemicals in the proper waste container.***

DATA ANALYSIS

1. Calculate the mass of water used for each trial of **Procedure 2**.

2. Using the densities of water in the table below, calculate the volume of water in the micropycnometer (and thus the volume of the micropycnometer itself) for each trial of Procedure 2. If the temperature of your water is not on the table, you can search for the density of water on the Internet.

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

3. Calculate the *average volume* for the micropycnometer. To determine the precision of your data, calculate the *standard deviation* for the volume of the micropycnometer as well. You may do this using *Excel*, your calculator functions, or by hand. For a description of various statistical functions as well as links to online manuals for various calculators to determine how to calculate them, go to the "Chemistry Statistics" link for the "Microscale Density" lab site.

   Please note that the number of significant figures in your average volume will be determined by the standard deviation of the volume, **not** by the significant figures in the numbers used to obtain the average volume. <u>Both the average and standard deviation are rounded off to the first significant digit of the standard deviation</u>. For example, if you have an average of 24.589341 with a standard deviation of 0.032985, they would *both* round to the first digit of the standard deviation and would be reported as 24.59 ± 0.03.

4. Calculate the mass of your unknown liquid used for each trial in **Procedure 3**. Using the average volume from Procedure 2, calculate the density of the unknown liquid for each trial of **Procedure 3**.

5. Calculate the average and standard deviation of the density of your unknown liquid.

6. Calculate the average and standard deviation of the refractive index of your unknown liquid from **Procedure 4**.

7. Compare the average density (d_4^{20}) and refractive index (n_4^{20}) for your unknown liquid to the densities and refractive indices in the table below to determine the identity of your unknown liquid.

Substance	d_4^{20} (g/mL)	n_4^{20}	b.p. (°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

POSTLAB ACTIVITY

You will be turning in a lab report. It may be an individual or group report, depending on instructor preference. The report should include the title, an abstract, results, discussion, and references. The information that you obtained from the data analysis should be included at some point in the report. It is up to you whether it is in the results or discussion or both. However, remember that the report is more than just answering some questions and that it should flow smoothly and logically as you discuss the data obtained, what it signifies, and potential errors or difficulties. Lab report guidelines for how to write the abstract, results, and discussion are found at http://webs.anokaramsey.edu/chemistry/Chem1061.

Follow your instructor's directions for submitting the report. If you are submitting electronically, please submit a single file with all of the required information. Use the following convention for naming your files: *Lastname1 Lastname2 Microscale* for a group report or *Lastname Firstname Microscale* for an individual report. If you are emailing the report, use a subject line of *Chem 1061: Microscale Lab*.

You will need to show sample calculations in the report. For electronic submissions, you may embed data tables which contain the formulas in calculated cells. (Ask your instructor to demonstrate how to do this.) For paper submissions, you will need to show these calculations for one trial of each procedure. You will also need to show these calculations if you submit the report electronically but do NOT include formulas in embedded data tables.