Objective
To identify a pure liquid substance using the physical properties of solubility, density, and boiling point.

Text reference
solubility, density, boiling point

Discussion
The physical properties of a pure substance can be measured without changing the composition of the substance. In this experiment you will learn techniques for determining solubility, density, and boiling point. These three physical properties will enable the determination of the identity of an unknown liquid by consulting a table of Physical Properties of Selected Pure Liquids (page 4).

I. Solubility
A liquid is soluble in another liquid if when the two are mixed—a clear solution results. Please note that *clear* liquids or solutions may still be *colored*; *clear* does not imply *colorless*. Cloudiness or the existence of droplets or layers indicates insolubility. One recognizes layers because of the presence of a meniscus that forms the boundary between the liquids. A meniscus may be very difficult to see, especially if both liquids are colorless and clear.

II. Density
Density is defined as mass per unit volume, which in equation form is written D = M/V. The typical units of density that a chemist encounters and uses are g/cm^3 or g/cc or g/mL. Because 1 mL = 1 cm^3 = 1cc, these three density units are equivalent. What does density fundamentally mean? Take for example a substance that has a density of 2 g/cm^3. If you had 1 cm^3 of this substance it would have a mass of 2 g. Or looked at from a different view, a sample of the substance that had a mass of 2 g would occupy a volume of space of 1 cm^3.

III. Boiling Point
When a liquid boils, the liquid changes from the liquid state to the gaseous state in the formation of bubbles. The bubbles of gas form within the liquid and rise freely to its surface and burst. The temperature at which this happens is called the boiling point or boiling temperature. The boiling point may be determined by observing the temperature of the gas (more generally referred to as a vapor in this situation) above a liquid while that liquid is boiled. The vapor surrounding the thermometer remains constant (at the boiling temperature) as the liquid boils.

Procedure

All procedures must be performed in the fume hood. When moving between hoods during density determination step, use the provided cap to cover your liquid!!

I. Solubility

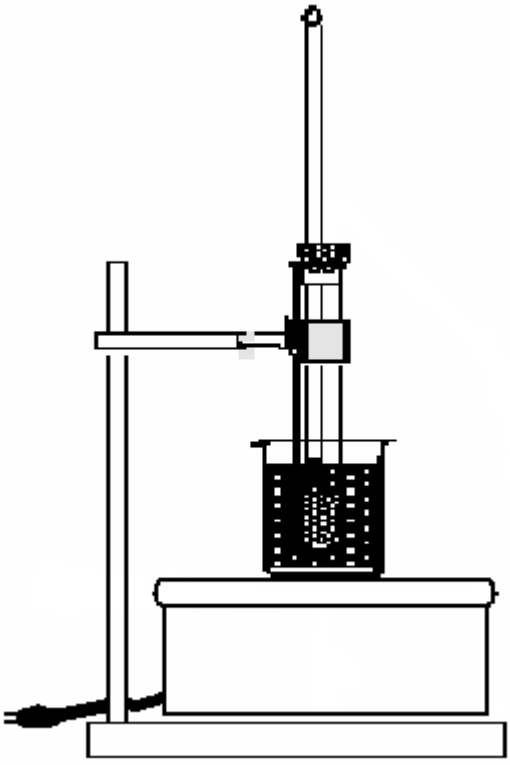
1. For this procedure, pay attention when your instructor demonstrates the *finger flick method* for mixing liquids without inverting the test-tube. Placing your finger over the top of the test tube and shaking is unacceptable laboratory technique!
2. Also, avoid cross-contamination of chemicals. Do not put the tip of the dropper inside the mouth of the test tube. Always put droppers back into their original container.
3. In a clean, dry test tube, combine **20 drops of your unknown** with **20 drops of ethyl alcohol**. If your unknown is soluble in ethyl alcohol, the two liquids should completely mix. If you unknown is insoluble in ethyl alcohol, you should be able to see two layers (the unmixed liquids) in the test tube. Record your observations.
4. Repeat the procedure in a new test-tube to determine the solubility of **your unknown liquid (20 drops)** in **water (20 drops**). Record your observations.
5. Dispose of the wastes from this section in the appropriate container and place your test tubes in the designated dirty glassware rack.

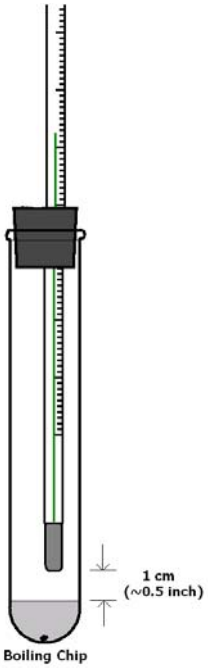
II. Density

1. Obtain a 10.00 mL graduated cylinder and determine its mass when empty. **You will use this same graduated cylinder for both trials. Therefore, use this same mass for both density trials. You should not reweigh the empty cylinder for trial 2.**
2. Dispense *about* five mL of your unknown liquid into the cylinder. You do NOT have to fill the liquid to exactly 5.00 mL. It is MORE IMPORTANT that you correctly read and record the true volume of the liquid to 2 places past the decimal.
3. Quickly and carefully reweigh the graduated cylinder with the added liquid.
4. Determine the mass of the unknown liquid sample.
5. You now have all the measurements necessary to calculate the density of the unknown liquid.
6. Pour the liquid from the graduated cylinder into the large test tube you will later use to determine the boiling point of your unknown liquid. Make sure your large test tube is properly resting in a medium beaker until incorporated into your boiling point apparatus. **You will have to move between hoods to do this – make sure to cap your cylinder when moving between hoods!**
7. Calculate the density of the unknown liquid.
8. Repeat steps 2-7 for a second trial. Use the same graduated cylinder you used in the first trial. Again, after the volume and mass measurements, pour the liquid from the graduated cylinder into the same large test tube.
9. **If the two density determinations *differ* by more than 0.03 g/mL, you must repeat the density determination with a third trial.**

III. Boiling point (see illustration below)

1. Place a 600mL beaker containing about 400mL of tap water on top of a hotplate in the fume hood.
2. You should already have *about* 10 mL of your unknown liquid stored in a large test tube (saved from the density determination).
3. *Put a piece of boiling chip into the unknown liquid to prevent bumping and to ensure even boiling.*
4. Clamp the test tube so that it is suspended in the water bath. Make sure the test tube does not touch the beaker and make sure that the level of liquid inside the test tube is below the level of water in the outer beaker; if not, adjust the height of the clamp.
5. Obtain a split-hole stopper and insert a thermometer into the *split hole*.
6. Turn the thermometer so that the scale can be read in the split. Doing this permits you to read the thermometer along its entire length.
7. Insert the stopper and thermometer into the test tube.
8. Adjust the thermometer so that its bulb is 1 cm above the unknown liquid (see the illustration below). Immerse the test-tube in the 600 mL beaker.





9. **Have your instructor check your setup and initial your report sheet before you begin heating.**

10. Turn the righthand dial on your hotplate to "6" or "High". Heat the water gradually (the beaker is a *hot water bath*) and watch for changes both in the heating water and especially in the test-tube. Be patient! When the liquid has reached its boiling temperature, you will observe a large number of boiling bubbles in the unknown liquid and the temperature should remain fairly stable. Record this temperature as the boiling point temperature. *Do not boil until the test tube is dry on the inside!* Boiling down to a small (or no) amount of your unknown liquid could lead to an inaccurate reading of its boiling point.

11. **Some liquids boil at temperatures much less than pure water. You must watch your unknown liquid constantly, to avoid evaporating *all* of your sample**!

12. Once you have determined the boiling point temperature, turn off the hotplate, remove the test-tube from the hot water bath and allow the assembly to cool.

IV. Disposal of Chemicals

Any “unknown liquid” poured out of the original vial should be disposed of in proper container in the hood. Vials containing unused liquid should be placed in the designated hood area. **No chemicals go into the sink.**

V. Identification of Unknown Liquid

Your unknown is one of the liquids in the following Table. Compare the density, boiling point, and solubilities of your unknown with those given in the Table below to determine the unknown liquid's identity. Select and indicate two liquids that most closely agree with the set of data you obtained.

Physical Properties of Selected Pure Liquids

Substance	Density (g/mL)	N. Boiling Temp. (°C)	Solubility in water	Solubility in alcohol
UNKNOWN ?				
acetone	0.79	57	soluble	soluble
cyclohexane	0.78	81	insoluble	soluble
ethyl acetate	0.90	77	insoluble	soluble
ethyl propyl ether	0.74	64	slightly	soluble
hexane	0.66	69	insoluble	soluble
methyl alcohol	0.79	65	soluble	soluble
n-pentane	0.63	36	insoluble	soluble
propanal	0.81	49	soluble	soluble
2-propanol	0.79	82	soluble	soluble

Report Sheets **ID a Liquid Report Sheets**

Name_____ Date_____ Lab Section_____

Record all observations and data in ink.
All measurements must have: quantity & units.

Unknown number # _____ **Balance number # _____**

I. Solubility

unknown liquid in water _____

unknown liquid in ethyl alcohol _____

II. Density
All measurements must have: quantity & units.

	trial 1	**trial 2**	**trial 3 (if necessary)**
volume of unknown liquid			
mass of cylinder + unknown liquid			
mass of empty graduated cylinder **(Use the same graduated cylinder for all trials. Measure the mass of the empty cylinder only once, then put that same value in all boxes in this row.)**			
mass of unknown liquid			
density of unknown liquid			

Density average of the 2 best trials....._____

III. Boiling point
All measurements must have: quantity & units.

Instructor's initials for correct experimental setup _____

boiling temperature of unknown liquid _____

IV. Identity of unknown liquid _____

2nd most likely candidate _____

Postlab Questions

1. Is it always possible to identify any unknown liquid correctly by density measurement only? Explain your answer.

2. a) What is the uncertainty associated with each of your volume readings? _____
(include the amount of uncertainty and the units)

b) True or False: There is no uncertainty associated with your mass readings, because they were measured directly by the balance.

c) *If* there **is** uncertainty associated with mass readings, what is the uncertainty associated with each of your mass readings? (include the amount of uncertainty and the units) _____

d) Although (due to uncertainty of measurements) you cannot know the exact mass of your empty graduated cylinder, you **do** know that it is very likely to fall between what two values?

_____

An empty graduated cylinder weighs 26.80 ± 0.01 g. The mass of the graduated cylinder with 10.0 ± 0.1 mL of liquid in it is 34.07 ± 0.01 g. What is the mass of the liquid?

If a graduated cylinder weighs 28.54 ± 0.01 g, and if the graduated cylinder containing 9.0 ± 0.1 mL of a liquid weighs 36.19 ± 0.01 g, what is the density of the liquid?

A student determines that an unknown liquid has a density of 0.78 g/mL, a boiling point of 63.0 ± 0.1 ^{o}C, and it is soluble in water and in ethyl alcohol. Use the table on page 4 to identify the most likely candidate for the unknown liquid. What is the second most likely candidate for the unknown liquid?

Do any of the unknown liquids boil at temperatures above the boiling temperature of pure water?

Where should you hold a thermometer when you want to insert it into a split-cork stopper?

List all of the precautions that must be taken when setting up and using the boiling point determination apparatus. Do not list "wear goggles." You must ALWAYS wear goggles when working with glassware and chemicals. Focus on safety and procedural precautions that are specific to the boiling point determination technique.