Experiment 2 - Using Physical Properties to Identify an Unknown Liquid

Introduction

Chemists use **properties** to identify the **substances** they encounter. Properties are the distinguishing characteristics of the materials, such as their color, phase (solid, liquid or gas), smell, etc. Substances are separated into several categories:

- **elements,** which contain atoms of only a single element (examples: aluminum metal, charcoal)
- **pure substances** or **compounds,** which contain only a single type of molecule, or a specific, consistent ratio of elements (examples: pure salt, sugar, and water)
- **mixtures,** which contain several different compounds (examples: air, milk, seawater)

Properties are also divided (somewhat arbitrarily) into **physical properties** and **chemical properties**. Generally, chemical properties are properties that have to do with reactions between substances, while physical properties do not involve reactions. For example, chemical properties include the ability to burn in air, or to dissolve in acid. Physical properties include density, color, hardness, boiling point, and melting point. Solubility (the ability to dissolve in another substance, for instance solid sugar dissolving in water to make syrup) is sometimes considered a chemical property and sometimes a physical property.

In this lab, you will assess the following properties of an unknown pure liquid:

- **boiling point** (the temperature at which the liquid boils, such as 100 °C for water)
- **melting point** (the temperature at which the liquid freezes, such as 0 °C for water)
- **density** (the ratio of mass/volume, usually measured in g/ml)
- **solubility** (or more correctly **miscibility**) in water (whether the liquid mixes completely with water, or forms two separate layers, like oil and water)

Your measurements will give you a possible range for most of the properties, rather than a precise number, but this is sufficient to determine which of the liquids in the table below you have. Specifically, we will measure the boiling point to within ±5 °C, whether is freezes above or below -78 °C and 0 °C, and if it is not soluble in water, whether it is more or less dense than water (density above or below 1 g/ml). After you measure these properties, you should be able to eliminate all but one of the liquids in the table, leaving only one possibility. It may be more helpful to eliminate impossible liquids rather than trying to match the correct one.

Safety Precautions:

- Wear your safety goggles at all times.
- Recommended: wear latex or nitrile gloves

Waste Disposal:

- Pour the used samples into the **organic waste** container in one of the fume hoods.

Physical Properties of some liquids

name of compound	formula of compound	boiling point (°C)	melting point (°C)	density (g/mL)	solubility in water
acetone	C_3H_6O	56	- 94	0.79	soluble
acetonitrile	CH_3CN	82	- 45	0.75	soluble
carbon tetrachloride	CCl_4	77	- 23	1.59	insoluble
cyclohexane	C_6H_{12}	81	+ 6	0.78	insoluble
cyclopentane	C_5H_{10}	49	- 94	0.75	insoluble
ethanol	C_2H_6O	78	- 115	0.79	soluble
methylene chloride	CH_2Cl_2	40	- 95	1.32	insoluble
t-butyl chloride	C_4H_9Cl	51	- 27	0.85	insoluble
"Freon-113"	$C_2Cl_3F_3$	48	- 35	1.6	insoluble
water	H_2O	100	0	1.00	N/A

Procedure

First obtain a sample of an unknown liquid. Record the unknown code on your report sheet. Three different properties of the sample will be measured: its solubility in water, its boiling point, and its melting point. You may do the three parts of the lab in any order.

Part 1 - Solubility in Water

Take an empty test tube and add enough water to it to fill approximately the bottom inch to two inches of the test tube. Then add a small portion of your unknown liquid to the test tube. You now have a mixture of two liquids. Stir or gently swirl the mixture to mix the two liquids. Does the unknown liquid dissolve in the water, or does it form a separate layer? If the unknown is **soluble** in water, it will completely mix with it to form a **homogeneous mixture**. The mixture will appear as one clear liquid, and will look the same throughout. If the unknown is **insoluble** in water, the mixture will be a **heterogeneous mixture** in which two separate phases are visible. The unknown will either float above the water or will sink below it to the bottom of the test tube, and you will be able to see a line in between the two layers.

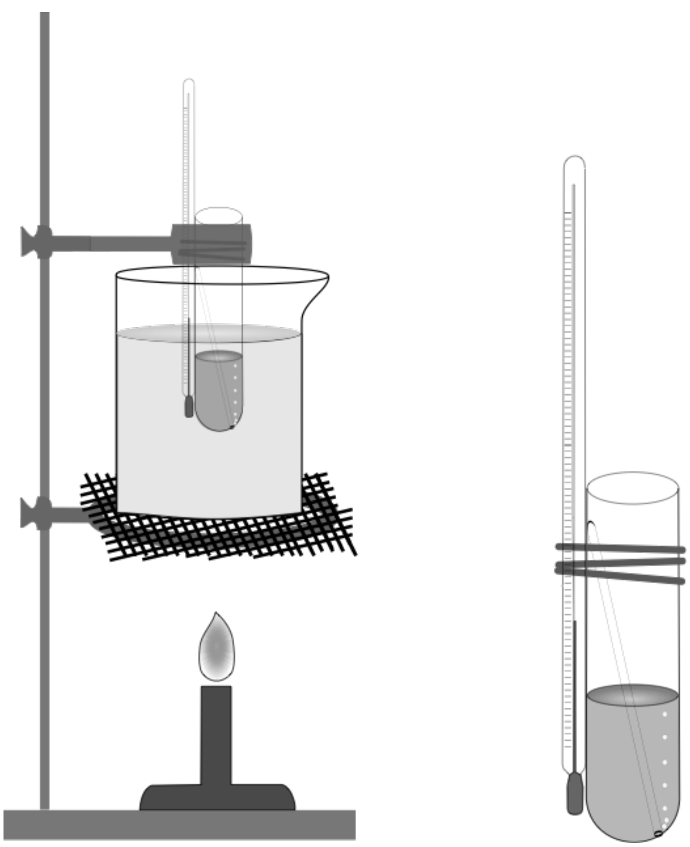
If the unknown liquid is insoluble in water, this test will also provide some information about another property of the liquid, its **density**. The density of a substance is the weight of a standard volume, usually one cubic centimeter (1 cc or 1 cm^3) of the substance. Density is a measure of the relative "heaviness" of a substance. If two liquids that are not soluble in each other are mixed together, the one with the greater density will sink to the bottom, and the one with the smaller density will float on top. Oil floats on water because the density of oil is less than that of water.

If your unknown was insoluble in water, add a few drops of copper sulfate ($CuSO_4$) solution. This blue solution will dissolve only in the water layer, and will color that layer blue, so you can tell which layer is water. The density of water is exactly 1.00 gram/cm^3. Any liquid that sinks when added to water has a density greater that 1.00 g/cm^3, and any liquid that floats on water has a density of less than 1.00 g/cm^3. Observing if the unknown sinks or floats will not tell you its exact density, but will only tell you if its density is greater than or less than 1.00 g/cm^3. If the unknown was soluble in water, its density cannot be estimated by this method. Whenever two liquids are soluble in each other, they mix evenly, regardless of their densities.

<u>Part 2 - Boiling Point</u>

The boiling point of the liquid will be measured by a microscale technique, which uses a very small amount of the liquid unknown. Refer to the following drawing when setting up the equipment for this part of the experiment.

Set up a ring stand with a water bath: attach a ring to the ring stand, put a piece of wire gauze on top of it, and then place a beaker of water on the wire gauze. Place about 0.5 mL of the unknown liquid in a small test tube, and place an empty capillary tube ***open end down*** into the liquid. Attach a thermometer to the small test tube using several rubber bands (stacked on top of each other). Make sure that the bulb of the thermometer is lined up with the level of the liquid inside the test tube. Clamp the assembly to the ring stand, submerging it into the water (make sure that the unknown liquid inside the small test tube is below the water level so that the unknown heats evenly, but do not let any water enter the test tube!).



Start heating the water bath using a Bunsen burner, and watch the unknown continuously. Keep heating the sample until a *rapid and continuous* stream of bubbles comes out of the small capillary tube. At this point, turn off the Bunsen burner. The bubbles will slow down, stop, and then some liquid will be drawn up into the capillary tube. Measure the temperature when the liquid enters the capillary: this is the boiling point of the liquid (within 5°C).

If your liquid has a low boiling point, it may evaporate completely from the test tube before you finish your measurement. If this occurs, repeat the measurement using a little bit more liquid in the test tube.

Variations in barometric pressure, impurities in the liquid, or difficulty choosing the correct moment to record the temperature may cause your measured boiling point to be a little different from the accepted boiling point listed in the table. Use your measured boiling point with its error range (± 5 °C) to eliminate liquids outside this range.

Part 3 - Freezing point

Most of the liquids on the chart do not freeze until they have been cooled to very low temperatures. Acetone, for example, only freezes if it has been cooled to –94 °C, or –137°F.

In this experiment, we will not measure the exact freezing temperature of the unknown liquids, but will only compare their freezing temperatures to the temperature of **dry ice** and ice-water. Dry ice is solid carbon dioxide (CO_2). It vaporizes to form gaseous CO_2 at –78 °C. While it is vaporizing, the solid dry ice maintains a temperature of –78 °C. A mixture of dry ice and acetone will be provided. The dry ice cools the acetone to –78 °C, but the acetone remains liquid because its freezing point is lower. An ice-water bath will also be provided; in this case, as the ice melts the bath maintains a constant temperature of 0 °C.

To test your unknown, take a dry test tube and add some unknown liquid to it. Add enough to fill the bottom one inch or so of the test tube. Hold the test tube with a test tube clamp in the dry ice–acetone bath. Leave the liquid totally immersed for at least two minutes. Remove the tube and check if the liquid has frozen. If the liquid appears to have partly frozen, immerse it in the dry ice–acetone bath again to see if it will completely freeze.

If the liquid freezes in the dry ice–acetone bath, its freezing point is higher than –78°C. If it does not freeze, its freezing point is lower than –78°C. If it does freeze in the dry ice–acetone bath, let it thaw, then immerse it in the ice-water bath and see if it freezes. This will tell you if the freezing point is above or below 0 °C. The actual freezing point of the liquid cannot be determined by this experiment.

Data Analysis

Use your results to eliminate all the impossible liquids in the table, leaving only one. The remaining liquid is your unknown. In the results section in your notebook, clearly indicate everything you know about the physical properties of your unknown and identify it by name.

Name: Section:

Experiment 2 Pre-Lab Sheet

Illustrate the possible range for each property on the number line, based on the experimental results given (do not use the data in the table for the ranges). Then identify the liquid from the list.

Sample 1: The boiling point is recorded as 43 °C, it does not freeze in the dry ice–acetone bath, and the sample is insoluble in water, with the blue layer above the clear layer.

Boiling point range (1pt):

0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 °C

Freezing point range (1pt):

-130 -120 -110 -100 -90 -80 -70 -60 -50 -40 -30 -20 -10 0 10 °C

Density range (1pt):

0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5 1.6 1.7 1.8 1.9 g/ml

Identity of sample 1 (2pts):

Sample 2: The boiling point is recorded as 80 °C, it freezes in the dry ice–acetone bath but not in the ice-water bath, and is soluble in water.

Boiling point range (1pt):

40 45 50 55 60 65 70 75 80 85 90 95 100 105 110 °C

Freezing point range (1pt):

-130 -120 -110 -100 -90 -80 -70 -60 -50 -40 -30 -20 -10 0 10 °C

Density range (1pt):

0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5 1.6 1.7 1.8 1.9 g/ml

Identity of sample 2 (2pts):