

Experiment 2 - Using Physical Properties to Identify an Unknown Liquid

We usually think of chemists as scientists who do things with chemicals. We can picture a chemist's laboratory with rows of bottles filled with different chemicals. Most of them are probably poisonous, explosive, or have terrible odors. But what are these chemicals, these elements and compounds with which a chemist works?

What we usually call a chemical can also be described as a substance consisting of only one kind of matter, a substance in which all the molecules are the same kind. As such, a chemical can also be described as a pure substance. Everything in the world consists of chemicals, but most objects that we find around us contain many different chemicals mixed together. These objects are not pure substances, but are mixtures of different pure substances. They contain different kinds of molecules mixed together.

Even though most common objects are thus classified as mixtures, they are all composed of molecules, and they are therefore all mixtures of different chemicals. Even such objects as trees, people, or bananas can be considered to be just very complicated mixtures of chemicals. The chemical components of mixtures such as these can be separated, and different pure substances can be isolated from them. For example, such pure substances as methyl alcohol (wood alcohol), acetic acid, cellulose, and sugar can all be obtained from trees.

Chemists have obtained literally millions of different pure substances, or chemicals, either by isolating them from natural sources or by synthesizing them from simpler chemicals. Each of these pure substances can be described by giving the formula of the molecules that it consists of (for example, water's formula is H_2O , salt's is $NaCl$, and sugar's is $C_{12}H_{22}O_{11}$), or by listing the physical properties of the substance.

The physical properties of a substance include all of the properties that are characteristic of that type of substance and that do not involve chemically converting the substance into another kind of substance (those properties are called chemical properties). The color of the substance, the temperature at which it melts or freezes (melting point), the temperature at which it boils (boiling point), the odor of the substance, and the solubility properties of the substance (does it dissolve in water or in other solvents?) are all physical properties. There are many more physical properties of substances that can be measured, but measuring some of these requires elaborate scientific instruments.

Every pure substance has its own unique set of physical properties. These may be similar to those of other pure substances, but they will not be exactly the same. Therefore,

if the identity of a pure substance is not known, it can be conclusively identified by observing and measuring its physical properties. The purpose of this experiment is to identify an unknown pure substance in this manner.

At the start of the experiment, you will be given a sample of a pure substance. Samples of several different pure substances will be available in the lab. All of these look the same - they are all colorless liquids at ordinary room temperature. Many of the physical properties of the substances are quite different, however, as can be seen on the chart below. This chart lists the names of all the pure substances that will be used in this experiment, along with the chemical formula of each one, and some of the physical properties of each one. In the experiment, you will obtain information about the properties of the substance you have been given. By matching the properties of your substance with those of the substances listed on the chart, you should be able to determine the identity of your unknown substance.

| name of compound | formula of compound | boiling point | melting point | density (in g/mL) | solubility in water |
|-------------------------|---|----------------------|----------------------|--------------------------|----------------------------|
| acetone | C ₃ H ₆ O | 56 °C | - 94 °C | 0.79 | soluble |
| acetonitrile | CH ₃ CN | 82 °C | - 45 °C | 0.75 | soluble |
| carbon tetrachloride | CCl ₄ | 77 °C | - 23 °C | 1.59 | insoluble |
| cyclohexane | C ₆ H ₁₂ | 81 °C | + 6 °C | 0.78 | insoluble |
| cyclopentane | C ₅ H ₁₀ | 49 °C | - 94 °C | 0.75 | insoluble |
| ethanol | C ₂ H ₆ O | 78 °C | - 115 °C | 0.79 | soluble |
| methylene chloride | CH ₂ Cl ₂ | 40 °C | - 95 °C | 1.32 | insoluble |
| t-butyl chloride | C ₄ H ₉ Cl | 51 °C | - 27 °C | 0.85 | insoluble |
| water | H ₂ O | 100 °C | 0 °C | 1.00 | N/A |
| "Freon-113" | C ₂ Cl ₃ F ₃ | 48 °C | - 35 °C | 1.6 | insoluble |

Safety Precautions:

- Wear your safety goggles at all times.

Waste Disposal:

- Pour the used samples into the **organic waste** container (which has a **pink** label) in one of the fume hoods.

Procedure

First obtain a sample of an unknown liquid. Record the unknown number 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

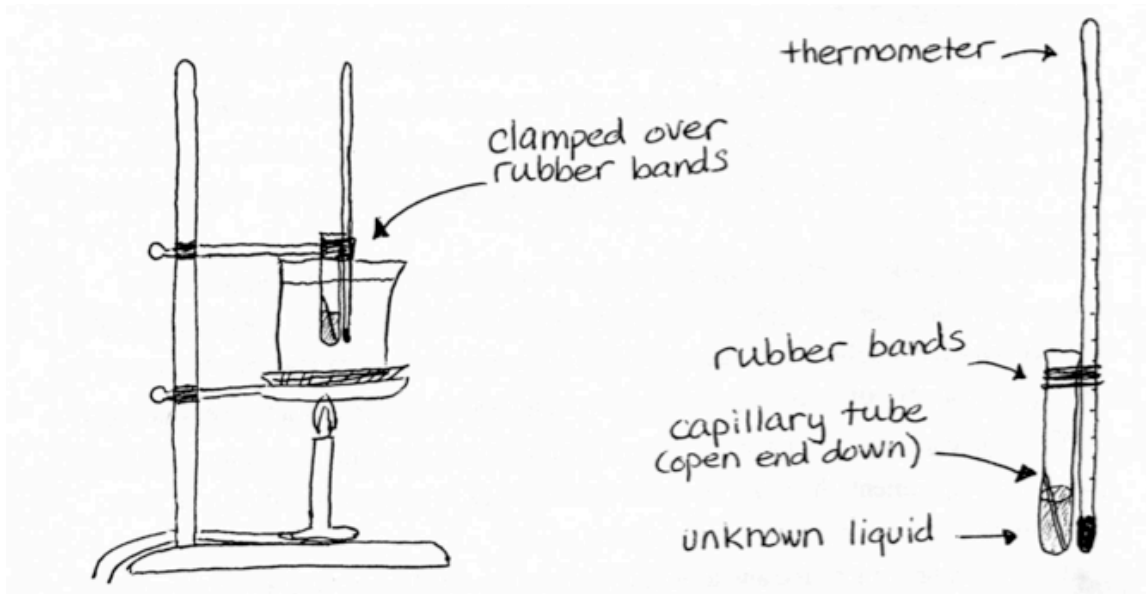
Take an empty test tube and add enough water to it to fill approximately the bottom inch to two inches of the test tube. Then add a small portion of your unknown liquid to the test tube. You now have a mixture of two liquids. Stir or gently swirl the mixture to mix the two liquids. Does the unknown liquid 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.

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 cm³) 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, did it float or sink when added to water? (If you are not sure whether the unknown is above or below the water, add a few drops of copper sulfate (CuSO₄) solution. This blue solution will dissolve only in the water layer, and will color that layer blue.) The density of water is exactly 1.00 gram/cm³. Any liquid that sinks when added to water has a density greater than 1.00 g/cm³, and any liquid that floats on water has a density of less than 1.00 g/cm³. 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/cm³. 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.

Part 2 - Boiling Point

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).

It is possible that the boiling point value you obtain may be a few degrees different from the value given on the chart. Variations in barometric pressure, impurities in the liquid, or an incorrectly calibrated thermometer may all contribute to this discrepancy.

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 degrees below zero Celsius. This temperature is -94°C , or -137°F . A few of the liquids, such as cyclohexane, have much higher freezing points.

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. Dry Ice is solid carbon dioxide (CO_2). It vaporizes to form gaseous CO_2 at -78°C (or -108°F). While it is vaporizing, the solid Dry Ice maintains a temperature of -78°C . It can cool any substance that comes in contact with it to this temperature. In this part of the experiment, the unknowns will be chilled to Dry Ice temperature, and their behavior at this temperature will be observed.

A mixture of Dry Ice and acetone has been prepared to provide a -78°C bath in which the unknowns can be immersed. 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 and immerse it in the Dry Ice-acetone mixture. Leave the liquid totally immersed for at least two minutes. Then remove the test tube. Has the liquid frozen? If the liquid appears to have partly frozen, immerse it in the Dry Ice-acetone mixture again to see if it will completely freeze.

If the liquid freezes at Dry Ice temperature, its freezing point is at a higher temperature than -78°C . If it does not freeze, its freezing point is at a lower temperature than -78°C . The actual freezing point of the liquid cannot be determined by this experiment.

Conclusion

You now have information about three or four of the physical properties of your unknown. Despite the fact that much of this information is not exact, you should now be able to determine which of the substances on the chart is your unknown. Which of the substances has physical properties that most closely match those of your unknown? This

can best be found by eliminating all substances that are significantly different from yours in any property. Keep in mind that the boiling point information is not very accurate. If your unknown is soluble in water, for example, all substances on the chart that are insoluble in water can be eliminated. If you found that your unknown was soluble in water, froze in Dry Ice, and boiled anywhere between 80°C and 85°C, this elimination process would show that it was acetonitrile.

Question

1. An unknown liquid was mixed with water, and two layers formed. When a blue solution of CuSO_4 was added and mixed, the top layer was colored blue. A fresh sample of the same unknown froze in dry ice, and the boiling point of the unknown was measured to be 82°C. What is the identity of the unknown? Clearly explain each step of your reasoning.