Identification of Substances by Physical Properties

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Purpose: To become acquainted with procedures used in evaluating physical properties and the use of these properties in identifying substances.

Equipment: Analytical balance, 50-mL beaker, 250-mL beaker, 25-mL container with screw cap, 10-mL graduated cylinder, large test tube, small test tubes, test tube rack, 8-mL pipet, ring stand, ring, wire gauze, no. 3 two-hole stopper with one hole slit to the side, Bunsen burner, rubber hose, stirring rod, glass beads, thermometer, microspatula, small watch glass, capillary tubes, glass tubing with right-angle bend, utility clamp, two-hole stopper, orthodontic-grade rubber bands, rubber bulb for pipet, corks

Materials: ethyl alcohol, cyclohexane, water, toluene, naphthalene, two unknowns (one liquid [vial #5], one solid [vial #5])

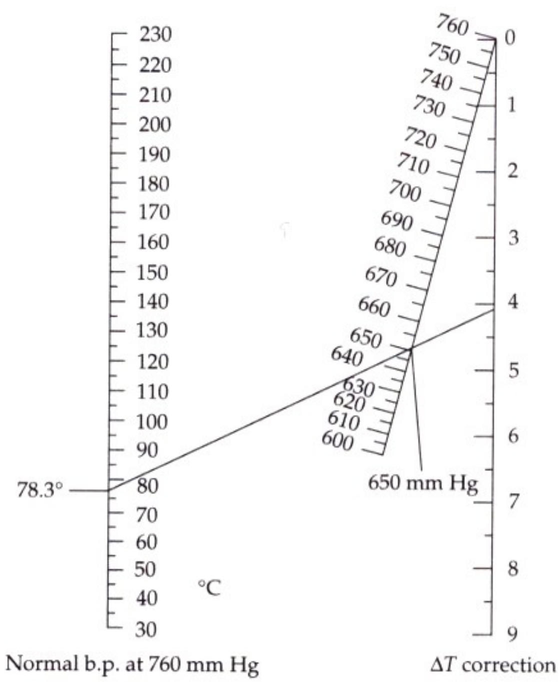
Introduction: Properties are those characteristics of a substance that enable scientists to identify it and distinguish it from other substances. Physical properties are those properties that can be observed without altering the composition of the substance. Some physical properties that can be observed quantitatively are melting point, boiling point, solubility, viscosity, density, and refractive index. A specific combination of properties is unique to a given substance, so it is possible to identify most substances just by careful determination of several properties.

The solubility of a substance in a solvent at a specified temperature is the maximum weight of that substance that dissolves in a given volume of a solvent. The solvents that will be used in this experiment are water, ethyl alcohol, and cyclohexane. Density is defined as mass per unit volume. These are two of the properties being measured in this experiment.

Melting points correspond to the temperature at which the liquid and solid states of a substance are in equilibrium. The melting point is the equilibrium temperature when approached from the solid phase.

A liquid is said to boil when bubbles of vapor form within it, rise rapidly to the surface, and burst. Any liquid in contact with the atmosphere will boil when its vapor pressure is equal to atmospheric pressure. Boiling points of liquids depend on atmospheric pressure. A liquid will boil at a higher temperature at a higher pressure or at a lower temperature at a lower pressure. The temperature at which a liquid boils at 760 mm Hg is called the normal boiling point.

Since atmospheric pressure is rarely exactly 760 mm Hg, a nomograph can be used to convert normal boiling point to boiling point at any pressure of interest. A nomograph looks like this:



▲ **FIGURE 2.5** Nomograph for boiling-point correction to 760 mm Hg.

To use a nomograph, simply line up the normal boiling point at 760 mm Hg with the pressure for the new measurement and find out how much to subtract from the normal boiling point. In the example above, the normal boiling point is 78.3°C. To find the boiling point at 650 mm Hg, simply subtract 4°C from the normal boiling point.

In the course of this experiment, a thermometer calibration curve will be used. This was constructed in a previous experiment. The observed freezing point was compared to the true freezing point, and the observed boiling point was adjusted for pressure and then compared to the true boiling point. The observed values were plotted against the true values. To use the calibration curve, one must interpolate data by drawing a line from the desired point on the "Observed" axis to the calibration curve, then down to the "True" axis. The value on the "True" axis under the spot on the curve corresponding to the "Observed" reading is the true value that should be reported.

In this experiment, two unknown solids will be identified using only the physical properties outlined above.

<u>Procedure</u>:

Part A – Solubility

1. Approximately 2 to 3 milliliters of each of ethyl alcohol, cyclohexane, and water were added to three clean, dry test tubes. A small sample of naphthalene was placed into each test tube. A cork was placed into each test tube and each test tube was shaken briefly. The cloudiness of the liquid was observed and recorded for each test tube.
2. Step 1 was repeated using the same solvents but a small sample of toluene instead of naphthalene.
3. Step 1 was repeated using the same solvents but a small sample of the solid unknown.
4. Step 1 was repeated using the same solvents but a small sample of the liquid unknown.

Part B – Density

1. About 1.5 grams of the solid unknown was weighed. The mass was recorded. A clean, dry 10-mL graduated cylinder was approximately half-filled with a solvent in which the solid unknown was insoluble. The volume of the solvent in the cylinder was measured and recorded.

2. The weighed solid was added to the liquid in the cylinder. The new volume was measured and recorded.

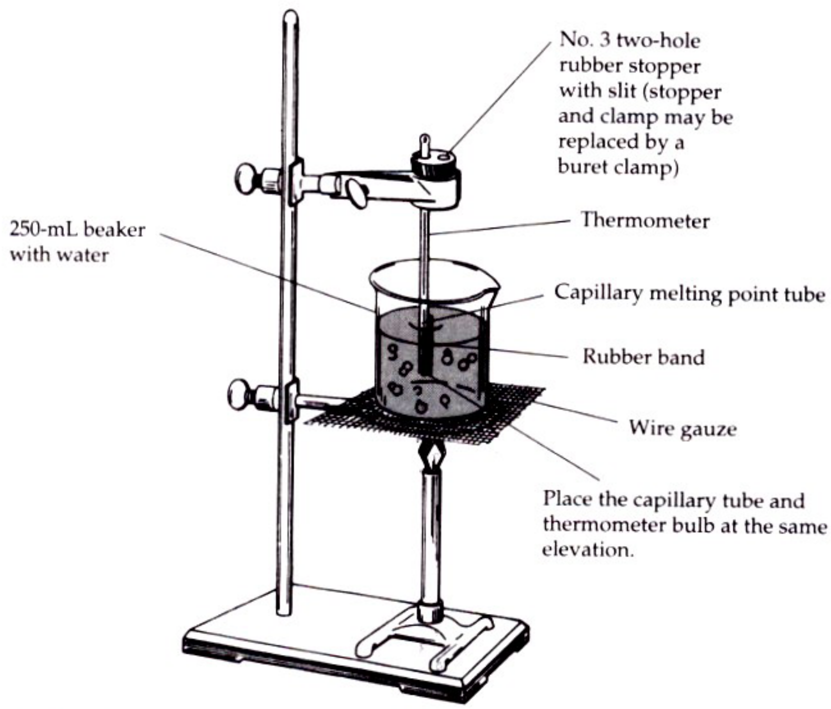
3. A container with a screw cap was weighed. Its mass was recorded.

4. 8 mL of the unknown liquid was transferred to the container via pipet. The container was then weighed. The mass was recorded.

Part C – Melting Point of Solid Unknown

1. A small portion of the solid-unknown sample was pulverized with the end of a test tube on a clean watch glass. The capillary tube was filled partially with the unknown by gently tapping the pulverized sample with the open end of the capillary to force some of the sample inside.

2. The melting point apparatus was set up as indicated in this diagram, using the marked parts in the indicated fashions:



▲ FIGURE 2.3 Apparatus for melting-point determination.

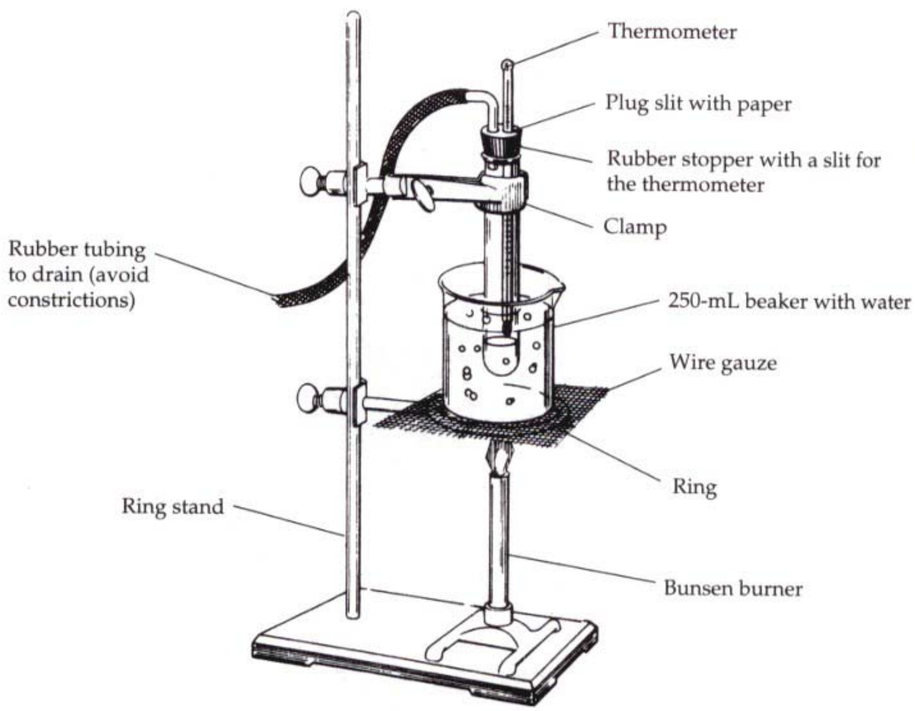
3. The rubber band was placed about 5 cm above the bulb on the thermometer, out of the liquid. The capillary tube was inserted under the rubber band with the closed end at the bottom. The thermometer with attached capillary was placed into the beaker of water so that the sample was covered by water.

4. The water was slowly heated while gently agitating the water with a stirring rod. At the moment the solid melted, the temperature was recorded. The melting point range was also recorded (the temperature range between the temperature at which the sample begins to melt and the temperature at which all of the sample has melted).

Part D – Boiling Point of Liquid Unknown

1. About 3 mL of the material used to determine the density of the liquid unknown was placed into a test tube. The test tube was fitted with a two-hole rubber stopper with one slit and one right-angle-bend glass tube. Two glass beads were dropped into the water.

The thermometer was positioned so that it was about 1 cm above the surface of the unknown liquid. The test tube was clamped in the ring stand. The right-angle-bend tubing was connected to a length of rubber tubing that reached the sink. The final apparatus looked like this:



▲ FIGURE 2.4 Apparatus for boiling-point determination.

2. The water was heated gradually. The temperature became constant at the boiling point of the liquid. The observed boiling point was measured and recorded.

Observations:

Part A - Solubility

Solubility of naphthalene: In water – insoluble; in cyclohexane – soluble; in alcohol – partly soluble

Solubility of toluene: In water – insoluble; in cyclohexane – soluble; in alcohol – soluble

Soluble of liquid unknown: In water – insoluble; in cyclohexane – soluble; in alcohol – soluble

Soluble of solid unknown: In water – soluble; in cyclohexane – insoluble; in alcohol – partly soluble

Part B – Density

Mass of solid weighed: 1.5008 ± .0001 g

Initial volume of liquid in cylinder: 5.0 ± .2 mL

Final volume of liquid in cylinder: 6.0 ± .2 mL

Volume of liquid: 8.0 mL

Mass of 25-mL bottle: 43.7031 ± .0001 g

Mass of 25-mL bottle plus 8 mL of unknown: 49.9113 ± .0001 g

Part C – Melting Point of Solid Unknown

Observed melting point: 60°C

Observed melting-point range: 53-63°C

Part D – Boiling Point of Liquid Unknown

Observed boiling point: 65°C

<u>Results</u>:

Volume of solid. (Final volume of liquid in cylinder *minus* initial volume of liquid in cylinder) =

6.0 ± .2 mL – 5.0 ± .2 mL =

1.0 ± .4 mL

Density of solid. (Mass of solid *divided by* volume of solid) =

1.5008 ± .0001 g / 1.0 ± .4 mL =

1.5008 ± .01% g / 1.0 ± 40% mL =

1.5008 ± 40.01% g/mL =

1.5 ± .6 g/mL

Volume of liquid corrected for the pipet correction. (Volume of liquid *plus or minus* previously ascertained uncertainty of pipet) =

8.0 ± .1 mL

Mass of liquid. (Mass of 25-mL bottle plus 8 mL of unknown *minus* mass of 25-mL bottle) =

49.9113 ± .0001 g – 43.7031 ± .0001 g =

6.2082 ± .0002 g

Density of liquid. (Mass of liquid *divided by* volume of liquid corrected for the pipet correction) =

6.2082 ± .0002 g / 8.0 ± .1 mL =

6.2082 ± 0% g / 8.0 ± 1.25% mL =

.776 ± 1.25% g/mL =

.78 ± .01 g/mL

Corrected melting point. (Observed melting point *interpolated on* attached thermometer calibration curve.) =

For interpolation markings, see attached curve.

~57°C.

Corrected melting-point range. (Observed melting point range *interpolated on* attached thermometer calibration curve) =

~51-59°C

Corrected boiling point: (Observed boiling point *interpolated on* attached thermometer calibration curve) =

~62°C

Solid unknown identification:

The solid unknown has a density of 1.5 ± .6 g/mL. It has a melting point of around 57°C. It is insoluble in water, soluble in cyclohexane, and partly soluble in alcohol. For these reasons, according to Table 2.1 on page 23 of *Chemistry: The Central Science Eighth Edition Laboratory Experiments*, the solid is identified as sodium acetate • $3H_2O$. This has a density of 1.45 g/mL. It has a melting point of 58°C. It is insoluble in awter, soluble in cyclohexane, and partly soluble in alcohol.

Liquid unknown identification:

The liquid unknown has a density of .78 ± .01 g/mL. It has a boiling point of around 62°C. It is insoluble in water, soluble in cyclohexane, and soluble in alcohol. It has a pungent odor. For these reasons, according to Table 2.1 on page 23 of the aforementioned source, the liquid is identified as chloroform. The densities do not match, but the rest of the properties do match.

Discussion: Clearly, if one of the properties in the liquid identification did not match, there must have been sources of error. Errors in measurement would have caused errors in calculations. If any liquid evaporated at any stage between measurements, then the measurements would have been inaccurate. If any material was lost during transfer at any stage, then measurements would have been incorrect. If the thermometer was not calibrated correctly in the original experiment, then all of the thermometric readings in this experiment would be incorrect. Since the beaker got cloudy during heating, the melting point of the solid sample was hard to observe. Therefore, the melting point might be different from what was observed. If the thermometer in the boiling point apparatus was allowed to touch the water, then the reading would have been incorrect. If the capillary tube was improperly secured to the thermometer in the melting point apparatus, then the reading might have been incorrect. If there was not enough of the solid in the capillary tube, then the reading may have changed due to lack of substance. Parallax view during the reading of graduated cylinders could have caused mistakes in interpretation of amount of substance in a cylinder.

The theory associated with this experiment is that the differences in properties between materials are due to the differences in bonding and structure. High melting and boiling points are results of molecular structure. Hydrogen bonds increase melting and boiling points incredibly. Ionic substances have higher melting points than molecular substances because of the types of bonds between particles. Furthermore, if any substance gives off an odor, it is because of weak bonding. If a solid has an odor, it is due to sublimation. If a liquid has an odor, it is because of rapid evaporation. The differences in properties between materials are intensive and are due to the differences in structure between materials.

The ramifications for this experiment are wide and far-reaching. The need to identify unknowns is extremely common, whether in industry, environmental research, the chemistry lab, or in any situation where some intensive properties are known, but not a name. Once a few intensive properties are known, all of the others can be figured out by deducing what the substance is. There are thousands of applications for the techniques learned in this experiment, and they are used frequently every day by people in a variety of fields.

<u>Questions</u>:

1. Thymol is a solid at room temperature.

2. Cyclohexane should be used to measure the density of magnesium nitrate.

3. A conversion from grams per milliliter to kilograms per liter would look like this: 1.5 g/mL (1 kg/1000 g)(1000 mL/1 L) = 1.5 kg/L

Grams per milliliter and kilograms per liter yield identical numbers.

4. If air bubbles were trapped in a solid beneath the liquid level, the volume measurement would be too high, and the calculated density would be too low.

5. A liquid unknown that was insoluble in water but soluble in cyclohexane and alcohol, and with a boiling point of 107°C at 658 mm Hg is toluene. To verify this, test the density, which should be .87 g/mL.

6. A liquid that has a density of 0.80 ± .01 g/mL that is insoluble in cyclohexane is propionaldehyde.

7. The boiling point of cyclohexane at 600 mm Hg is 87.5°C.

8. The density of osmium at 293 K is 22.4 g/cm^3. Its melting point is 3045°C. (source: http://www.chemicalelements.com/elements/os.html)

9. $MnSO_4$ is pale pink. It is very soluble in cold water. (source: http://soilchem.ag.ohio-state.edu/webdoug/MSDS2/manganese%20sulfate.pdf)

10. The formula of lead azide is $Pb(N_3)_2$. Its molar mass is 291.24 grams per mole. It is cream-colored. (source: http://roguesci.org/megalomania/explo/lead_azide.html)

Conclusion: The experiment was successful to a reasonable degree of success. Procedures used in evaluating physical properties were learned, and they were used to identify substances.