1. Identification of a Substance

Objective

Selected physical properties of an unknown substance will be measured experimentally and compared with the tabulated properties of known substances.

Introduction

Modern instrumental methods permit the routine analysis and identification of unknown substances. Because of the high cost of precision instruments, and due to the cost and time required for maintenance and calibration of such instruments, however, instrumental methods of analysis are primarily used for *repetitive* determinations of *similar* samples, in which case the instrumental method is relatively fast and the cost *per analysis* moderate. For a *single* sample, however, it is found that classical "wet" laboratory methods of analysis are often preferred.

Most commonly, a thorough determination of the *physical properties* of a sample will suffice for an identification. The properties determined for the *unknown* sample are compared to the properties of *known* substances as tabulated in the chemical literature. If the properties match, an identification is assumed. The physical properties most commonly used in identifications are *density* (as discussed in experiment "Density Determinations"), the *boiling and melting points* (as discussed in experiments "The Determination of Boiling Point" and "The Determination of Melting Point"), and the *solubility* of the substance in water or in some other appropriate solvent (as studied in experiment "The Solubility of a Salt").

In general terms, one substance is likely to be soluble in another substance if the two substances have similar structural features (for example, a similar group of atoms) or have comparable electronic properties (which lead to similar interparticle forces). Generally solutes are divided into two major classes, depending on whether they dissolve in very *polar* solvents like water, or in very *nonpolar* solvents such as any of the hydrocarbons (the hydrocarbon cyclohexane is used in this experiment). Generally ionic and very polar covalent substances will dissolve in water, whereas very nonpolar substances will dissolve in cyclohexane. Some solutes that are intermediate in polarity may dissolve well in a solvent of intermediate polarity (such as ethyl alcohol).

The physical properties discussed above are almost always reported in the literature when new substances are prepared, and are tabulated for previously known substances in the various handbooks of chemical data found in most laboratories and science libraries. Other gross properties that may also be helpful in identifications include unusual *color* (e.g., for transition metal ions of characteristic color), *odor* (e.g., some types of chemical substances have very characteristic odors), *hardness* (e.g., crystals of ionic substances are very hard compared to most covalently bonded substances), and so forth.

In this experiment, you will be provided with an unknown sample chosen from those substances listed in Table 1-1. By careful determination of the physi-

cal properties of your sample, you should be able to match those properties to one of the substances listed in the table. The unknown samples have been purified and contain only a single substance. In real practice, an unknown sample may contain more than one major component and is likely also to contain minor impurities. The identification of unknown samples is a vital, important, and interesting application of modern chemistry.

Table 1-1. Physical Properties of Selected Substances

Liquid Substances (under normal room conditions)

Substance	***Solubility***			***Density***	***Boiling Point***
	W	*A*	*C*	g/mL	°C (760 mm)
acetone	s	s	s	0.791	56
1-butanol	s	s	s	0.810	117
1-butanol, 2-methyl	s	s	s	0.816	128
2-butanol	s	s	s	0.808	100
2-butanol, 2-methyl	s	s	s	0.806	102
cyclohexane	-	s	s	0.778	81
ethyl acetate	s	s	s	0.900	78
hexane	-	s	s	0.660	68
methanol	s	s	s	0.793	65
1-propanol	s	s	s	0.780	97
2-propanol	s	s	s	0.786	83
2-propanol, 2-methyl	s	s	s	0.789	82

Solid Substances (under normal room conditions)

Substance	***Solubility***			***Density***	***Melting Point***
	W	*A*	*C*	g/mL	°C
acetamide	s	s	-	1.16	82
acetanilide	-	s	s	1.22	113
benzoic acid	-	s	s	1.27	123
benzophenone	-	s	s	1.15	48
biphenyl	-	s	s	0.867	71
camphor	-	s	s	0.990	176
1,4-dichlorobenzene	-	s	s	1.25	53
diphenylamine	-	s	s	1.16	53
lauric acid	-	s	s	0.867	44
naphthalene	-	s	s	1.03	80
phenyl benzoate	-	s	s	1.24	71
sodium acetate·$3H_2O$	s	s	-	1.45	58
stearic acid	-	s	s	0.941	72
thymol	-	s	s	0.925	52

Solubility legend: *W* = water, *A* = ethyl alcohol, *C* = cyclohexane
s = soluble, - = insoluble (or very low solubility)

SAFETY PRECAUTIONS

- **Wear safety glasses at all times while in the laboratory.**
- **Many of the unknown substances in this experiment are *flammable*; keep the unknown substances away from open flames.**

- **Cyclohexane, ethyl alcohol, acetone, hexane, methanol, ethyl acetate, and propanol are all highly flammable; keep these solvents in the exhaust hood.**
- **Many of the unknown substances in this experiment are *toxic* if inhaled or absorbed through the skin.**
- **Oil is used as the fluid in the Thiele tube used for boiling/melting point determinations. The oil may spatter or ignite if heated too quickly or to a temperature above 200°C. Examine the oil for contamination before use.**

Apparatus/Reagents Required

Unknown sample, distilled water, ethyl alcohol, cyclohexane, sodium chloride, copper(II) sulfate, pentane, oleic acid, naphthalene, melting/boiling point apparatus (oil-filled Thiele tube), melting point capillaries, rubber bands, semimicro test tubes, thermometer

Procedure

Record all data and observations directly in your notebook in ink.

Obtain an unknown substance for identification, and record its code number in your notebook and on the report page.

Record your gross observations of the color, odor, physical state, volatility, viscosity, clarity, and so forth for the unknown substance.

A. Solubility

Before determining the solubility of your unknown sample, you will perform some preliminary solubility studies with known solutes and several solvents.

Set up three clean, dry semimicro test tubes in a test tube rack. Place 10 drops of distilled water in one test tube, 10 drops of ethyl alcohol in a second test tube, and 10 drops of cyclohexane in the third test tube.

Obtain a sample of sodium chloride, and transfer approximately equal small amounts to each of the test tubes containing the three solvents. Stopper the test tubes and shake for at least 30 seconds to attempt to dissolve the solid. Record your observations on the solubility of NaCl in the three solvents.

Repeat the solubility tests with samples of naphthalene, copper(II) sulfate, pentane, and oleic acid, determining the solubility of each solute in each of the three solvents (water, ethyl alcohol, cyclohexane). Some of the test solutes are liquids. Add 5 drops of the liquid solute to each of the test tubes containing the solvents. If a liquid does not dissolve in a given solvent, it will form a separate layer in the test tube. Record your observations of solubility.

Repeat the solubility test, using small samples of your *unknown* material in each of the three solvents. Record your observations.

B. Density

Techniques for the measurement of the densities of solids and liquids were discussed in detail in experiment "Density Determinations." Review the discussion given there.

If the unknown is a liquid, determine its density by measuring the mass of a specific volume of the liquid. For example, a weighed empty graduated cylinder could be filled with the liquid to a particular volume and then reweighed. Alternatively, a specific volume of liquid can be taken with a transfer pipet, and the liquid weighed in a clean, dry beaker.

If the unknown is a solid, the volume of a weighed sample of solid may be determined by displacement of a liquid in which the solid is *not soluble* (see Part A of this experiment). If this is performed in a graduated cylinder, the change in liquid levels reflects the volume of the solid directly.

C. Melting and Boiling Point Determinations

Semimicro methods for the determination of boiling and melting points were discussed in detail in experiments "The Determination of Boiling Point" and "The Determination of Melting Point". Review these methods.

If your unknown sample is a solid, determine its melting point as you did in experiment "The Determination of Melting Point." Fill two or three melting point capillaries with finely powdered solid to a height of about 1 cm. Attach one of the capillaries to a thermometer with a rubber band, and lower the sample and thermometer into the oil bath of a Thiele tube. Heat the oil bath in such a manner that the temperature rises only one or two degrees per minute, and watch carefully for the crystals to melt. Record the melting *range* if the crystals do not melt sharply at a single temperature. Repeat the determination until you get two values that check to within one degree.

If your unknown is a liquid, determine its boiling point. Fill a micro test tube to a height of 3–4 cm with the liquid, and insert a short length of melting point capillary as described in experiment "The Determination of Boiling Point." Attach the test tube to a thermometer with a rubber band, and lower into the oil bath of a Thiele tube. Heat the oil bath until bubbles come in a steady stream from the capillary, remove the heat, and record the exact temperature at which bubbles stop coming from the capillary. Add liquid to preserve the 3–4-cm height, add a fresh capillary tube, and repeat the determination as a check. If your results do not agree within one degree, perform a third determination.

Compare the properties determined for your unknown with the properties of those substances listed in Table 1-1. Make a tentative identification of your unknown substance.

Identification of a Substance

Date: ..... Student name: .....

Course: ..... Team members: .....

Section: .....

Instructor: .....

Prelaboratory Questions

1. Why are the freezing point of a liquid and the melting point of a solid the same temperature for a given substance?

2. Familiarity with the various handbooks of chemical and physical data is very important in the identification of unknown substances. Using such a handbook, find information about the melting point, boiling point, density, and qualitative solubility of each of the following substances. Give your reference, including the page of the reference on which the data can be found.

Salicylic acid Reference ..... Page .....

Melting point ..... Density .....

Boiling point ..... Solubility .....

2-Pentanone Reference ..... Page .....

Melting point ..... Density .....

Boiling point ..... Solubility .....

1,2-Dibromomethane Reference ..... Page .....

Melting point ..... Density .....

Boiling point ..... Solubility .....

Isopropyl acetate Reference ..... Page .....

Melting point ..... Density .....

Boiling point ..... Solubility .....

3. A common mistake many students make is to confuse *whether or not* a solute is soluble in a solvent with how *quickly* a solute dissolves. Use your textbook to find a specific definition of solubility. What factors affect the solubility of a solute in a solvent? What factors affect the rate at which a solute dissolves in a solvent?

Identification of a Substance

Date: ..... Student name: .....

Course: ..... Team members: .....

Section: .....

Instructor: .....

Results/Observations

Unknown identification number .....

Qualitative observations (color, etc.) of unknown

A. Solubility Test Observations

Solubility of known solutes observations

Solubility of unknown observations

B. Density Determination

Describe the method by which the density of the unknown was determined. .....

.....

Density of unknown .....

C. Melting/Boiling Point Determination

Describe the method used for the melting or boiling point determination. .....

.....

Melting or boiling (circle) point determined .....

Barometric pressure (for boiling point) .....

Summarize in the following table the properties determined for your unknown sample, along with the properties of the substance you believe your unknown to be. Calculate the percentage difference between the known and unknown substance's properties for each quantitative determination.

Substance unknown is believed to be .....

	Unknown	Known	% difference
Solubility	.....	.....	.....
Density	.....	.....	.....
Melting/boiling point	.....	.....	.....

Questions

1. What *error* is introduced into a melting point determination if the oil bath is heated too quickly? Will the observed melting point be too high or too low?

2. In this experiment you qualitatively determined whether or not a given solute would dissolve in each of three solvents. Suggest a method by which the solubility of a solute in a solvent might be measured on a *quantitative* basis.