

Experiment 2: What Is It? Identifying an Unknown Compound

Pre-Laboratory Assignment

1. Outline the procedure which should be followed for testing the solubility of a compound.
2. Propose a procedure for determining the *density* of a solid compound.
3. Explain how to test the acidity/basicity of a solution using litmus paper. Describe another test that can be used to determine the acidity or basicity of a compound.
4. Make a table in your notebook that you will use to record your observations.

Objective: In this experiment, you will identify an unknown, solid, white compound by comparing the results of careful observations and tests that you make on the unknown to observations and tests made on a series of standard compounds. You will work with a team to characterize the standards.

Introduction

When Jason's great uncle Frederick passed away, his parents asked him to help out by sorting through the items that Uncle Fred had stored away in his attic. In a dusty corner behind an old chair, Jason found an antique apothecary chest with nine stoppered bottles inside. Each bottle contained a white powder, however, all the labels had fallen off of the bottles. Jason found the labels in a small pile at the bottom of the chest. Excited about his find, Jason immediately made plans to take the apothecary chest to "The Antiques Roadshow", which was due to visit nearby Providence in three weeks, to have it appraised. He felt that it would make a more impressive showing if the labels were actually affixed to the bottles. Being a perfectionist, he wanted to put the correct labels on the correct bottles, but all of the powders looked so similar that their appearance did not provide a clue as to their identities. The names on the labels were relatively common materials that he could find in his own home or easily obtain in a pharmacy, grocery or hardware store: baking soda, aspirin, corn starch, an ingredient found in fertilizer, caffeine, chalk, caustic lime, Epsom salt and borax. He decided to perform some tests to identify each of the nine substances.

The experiment

In today's experiment, you and the members of your group will perform the same tests that Jason has to carry out in order to solve his dilemma. The nine "known" compounds, or *standards*, will be provided for you. Work in a group of three or four people to fully characterize these standard compounds. Each student should characterize two or three standards. In addition, each *student* will be given a test tube that contains a solid, white compound that is one of the nine substances found in Uncle Fred's chest. You are responsible for identifying and fully characterizing this unknown compound. Record the code number of your test tube in your lab notebook.

Tests and Observations

The following are tests and observations that you should make on your unknown and on the standards that you characterize:

1. Observations of the physical appearance of the compound.

2. Solubility of the compound

When a solid compound dissolves in a particular liquid, we say that the compound is *soluble* in that particular *solvent*. If a compound does **not** dissolve in a particular solvent, we say that it is *insoluble*. If **some** of a compound dissolves in the solvent, we say it is *slightly soluble*. In quantitative terms, the following ranges for number of milligrams of solid which dissolve per milliliter of solvent can be used to define the terms:

>30 mg/mL **soluble** (a significant amount dissolves)
 <10 mg/mL **insoluble** (no detectable amount dissolves)
 10-30 mg/mL **slightly soluble** (a moderate amount dissolves)

These limits are, however, approximate and the solubility of a solid compound is usually determined quickly and easily in the lab in the following manner:

Place a "spatula-tip" amount of the compound in a small test tube. You can look at the samples of 0.010 g of solid that are provided in the lab, to get an idea of how much should be used. Add about 10 drops of the solvent to the test tube and agitate the contents by shaking the bottom of the test tube back and forth for a minute or so. Observe whether or not any of the solid dissolves.

3. Density

The density of a compound is an *intrinsic property*, which means that it is a characteristic property of the compound that can be used to help identify it. As you know from Experiment 1, density can be determined by measuring the mass and volume of a sample and dividing:

$$d = m/V, \quad \text{where } d = \text{density in g/mL; } m = \text{mass in g; } V = \text{volume in mL}$$

With your teammates, design a procedure for determining the density of a solid compound. Check this procedure with your TA before beginning.

4. Acidity/Basicity

Acids are compounds which:

- taste sour
- react with active metals (such as zinc and iron) to dissolve the metal and produce hydrogen gas
- react with bases to form water and ionic compounds called *salts*
- can donate a hydrogen ion

Bases are compounds which:

- taste bitter
- feel slippery or soapy on the skin
- react with acids to form water and a salt
- can accept a hydrogen ion

You will learn about acids and bases in great depth later in the course.

Both acids and bases are corrosive materials that should **never** be tasted in the laboratory and should not come into contact with your skin. They have an additional property wherein they will react with certain dyes known as *acid-base indicators*. A very common acid-base indicator is called *litmus*. An **acid** will react with litmus to produce a **red** color, while the reaction of a **base** with litmus produces a **blue** color. Some compounds are neither acidic nor basic and are referred to as *neutral* compounds. These compounds will not change the color of neutral litmus paper. Thus, a simple and safe test for determining the acidity or basicity of a solid compound is to dissolve it in water and test the resulting *solution* with litmus paper. **Note:** Make sure that you have "*neutral*" litmus paper in your drawer, not "red" or "blue". If you do not have the correct paper, trade it in at the stockroom.

The proper technique for testing a solution with indicator paper is to remove a drop of the solution with a stirring rod and place it on a piece of the paper. Do not place the paper directly into the solution being tested. Be sure to rinse off the stirring rod with water before placing it in the next solution you test. Before testing any of the compounds, place a drop of water, a **neutral** compound, on a piece of neutral litmus paper. The color of the paper in water provides you with a reference against which you can compare the color of the paper that results from other tests.

In addition to the litmus test, and especially if the compound is insoluble in water, it is useful to observe its behavior in acidic and basic solutions. If the compound is acidic, it will react with a solution of a base, such as sodium hydroxide, NaOH. If it is basic, it will react with a solution of an acid, such as hydrochloric acid, HCl. Evidence that a reaction has occurred can include dissolution of the solid, evolution of a gas (bubbles) or generation of heat. A neutral compound may or may not react with an acid or base. Compounds that have both acidic and basic properties also exist and are called amphoteric.

To test how a compound interacts with an acid or a base, place a "spatula-tip" amount in a test tube. Add about 10 drops of HCl solution or 10 drops of NaOH solution to the test tube, and agitate the contents of the tube by shaking the bottom of the test tube back and forth for a minute or so. Record your observations.

Using these tests and observations, develop an experimental procedure that will allow you to identify and characterize your unknown compound. Do not use more than 2 g of your unknown compound for the tests. Return the unused portion in the test tube to your TA. Do not contaminate the contents of this test tube.

The following resources will be provided for you:

Standards (each group should take no more than 2 g of each of the following):

- baking soda
- aspirin
- corn starch
- fertilizer ingredient
- caffeine
- chalk
- caustic lime (**corrosive!**)
- Epsom salt
- borax

Solvents (each group should take no more than 50 mL of each of the following):

acetone
ethanol
hexane
don't forget water!

10% aqueous solution of hydrochloric acid, HCl (50 mL per group)

10% aqueous solution of sodium hydroxide, NaOH (50 mL per group)

Note: a 10% solution contains 10 g of the compound dissolved in 90 g of the solvent (water, in this case).

Equipment: 10 mL graduated cylinder with a precision of ± 0.02 mL (1 per student)

Precautions:

Hydrochloric acid, HCl, and sodium hydroxide, NaOH, are both corrosive chemicals. If either of these solutions comes into contact with your skin, rinse it with copious amounts of water. Some of the unknowns are also corrosive.

Always assume that an unknown compound is toxic and potentially dangerous and use the proper precautions.

Acetone, hexane and ethanol (organic solvents) are all **flammable**. Keep away from open flames and heat sources!

Do **not** put any remaining liquid or solid material back into the dispensing jars as this could lead to contamination. Share it with another group or dispose of it in the proper manner. Also, do not contaminate the unknown compound contained in the test tube as the unused portion must be returned to your TA.

Important information about chemical waste from this experiment!

All of the solid compounds are non-hazardous and may be disposed of down the sink when dissolved in **water**, **HCl** or **NaOH** solutions. The three solvents which are provided, **acetone** (including "**wash**" **acetone**), **ethanol** and **hexane**, and any solutions that contain these solvents, *must* be disposed of in the **Organic Laboratory Byproducts** jar.

Questions

1. Describe the procedure that your team followed in performing this experiment and indicate what your role was in carrying out this procedure (what tasks you performed and which observations were yours).
2. Write the code number that was on your unknown. What is the identity of your unknown? What results prompted you to arrive at this conclusion? List the characteristics of your unknown.
3. Discuss sources of error associated with the procedure your team used for determining the density of a solid compound and the effects these errors might have on the results.