1. Identification of an Unknown Compound

Introduction

Chemistry is a science that is built on the interrelationship of experiment and theory (Ebbing/Gammon, Section 1.2). Experiments have led to theories, and theories have, in turn, led to other experiments. Accurate and complete observations are required in these experiments to provide a maximum amount of useful information. Without good observations, the cyclic relationships between experiment and theory would be seriously marred and perhaps destroyed.

When a student observes an event in the laboratory, it is not necessarily true that he or she will record a complete and accurate description of that event. Good observations require practice and attention to detail.

Purpose

This experiment emphasizes the importance of observations and the inferences that can be drawn from those observations. You will be able to identify an unknown compound by comparing its reactions with those of some known compounds. Accurate, complete observations are the only requirement for success. No knowledge of the chemistry that you will observe is necessary or assumed.

Concept of the experiment

You will obtain solid samples of sodium chloride (NaCl), sodium iodide (NaI), sodium carbonate (Na_2CO_3), sodium hydrogen phosphate (Na_2HPO_4), and sodium sulfate (Na_2SO_4), as well as solutions of these compounds in water. You will test these compounds with solutions of nitric acid (HNO_3), barium nitrate [($Ba(NO_3)_2$], silver nitrate ($AgNO_3$), and an acid–base indicator called thymol blue. Simultaneously, you will test an unknown solid sample that is identical to one of the known solid samples. You will be able to determine its identity by matching its characteristic reactions with those of the known compounds.

Three important reaction signals

In this experiment, certain signals will indicate that chemical reactions have taken place:

1. The color of a solution changes.
2. A gas evolves from the solution.
3. A precipitate appears or disappears.

The first of these signals is easy to interpret and use, but the other two signals require a little more explanation.

When gases are evolved from a solution, you will see bubbles form and move upward through the solution. If you see only two or three bubbles, gas evolution has not occurred. When a gas is evolved during this experiment, it will be very noticeable.

Precipitates are solids, but they may be so finely divided that they appear milky. An example of the formation of a precipitate is shown in Figure 1.1. Gradually, this precipitate will settle to the bottom of the test tube.

Procedure

Getting started

1. Obtain the unknown compound from your laboratory instructor.
2. You will also need 6 test tubes, a medicine dropper, and red and blue litmus paper.
3. Obtain directions from your laboratory instructor for discarding the solutions that you will use in this experiment.

Testing for gas evolution

1. Mark your test tubes for recognition with a marking pencil.
2. Use a clean spatula to place pea-sized solid samples of NaCl, NaI, Na_2CO_3, Na_2HPO_4, Na_2SO_4, and your unknown in the test tubes.
3. Add 5 drops of the solution of HNO_3 to each test tube and record your observations.

CAUTION: Nitric acid can cause chemical burns in addition to ruining your clothing. If you spill any of this solution on you, wash the contaminated area thoroughly with tap water and report the incident to your laboratory instructor. You may require further treatment.

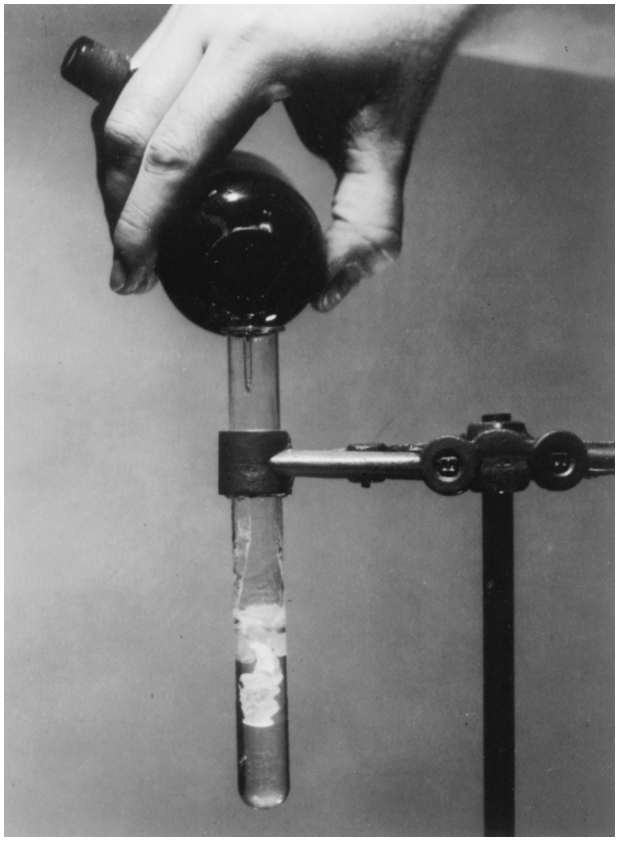

FIGURE 1.1
The formation of a cloudy precipitate when one solution is added to another.

4. Discard the solutions in the test tubes.
5. Wash the test tubes and rinse them with distilled water.

Dissolving your unknown compound

1. Wash a 100-mL graduated cylinder and a 400-mL beaker thoroughly and rinse them with distilled water.
2. Place another pea-sized portion of your unknown in the beaker. Add 200 mL of distilled water from the graduated cylinder, and swirl or stir gently until all the solid has dissolved.
3. Set aside the remaining portion of your unknown compound in a safe place for use in the event of an unforeseen accident.

Testing with barium nitrate

1. Use the solutions of NaCl, NaI, Na_2CO_3, Na_2HPO_4, and Na_2SO_4 that you will find in the laboratory and the solution of your unknown compound that you have just prepared for the remaining tests in this experiment.
2. Place 20 drops of the solution of NaCl in a clean, correctly marked test tube. Add 3 drops of a solution of ammonia (NH_3). Use a clean, dry stirring rod to stir the solution. Remove the stirring rod and touch the adhering drop of solution to a small piece of red litmus paper. If the paper does not turn blue, add drops of NH_3 to the solution in the test tube, with stirring, until it does.

   CAUTION: Ammonia can cause chemical burns in addition to ruining your clothing. If you spill any of the solution on you, wash the contaminated area thoroughly and report the incident to your laboratory instructor. You may require further treatment.

3. Repeat Step 2 with each of your solutions.
4. Add 5 drops of the solution of $Ba(NO_3)_2$ to each test tube. Shake gently to obtain homogeneity. Do not use your finger as a stopper. Examine each test tube carefully and look for precipitates. Record your observations, noting the color of each precipitate.

   CAUTION: Wash your hands thoroughly after using the solution containing barium because it is poisonous.

5. The test in this step applies only to those test tubes that contain precipitates. Add 10 drops of the solution of HNO_3 to each of these test tubes. Test each solution with blue litmus paper, using a clean, dry stirring rod. If the paper does not turn pink, add drops of the solution of HNO_3 to the solution in the test tube, with stirring, until it does. Examine each test tube. Which precipitates have dissolved? Record your observations.
6. Discard the solutions in the test tubes.
7. Wash the test tubes and rinse them with distilled water.

Testing with silver nitrate

1. Use fresh solutions for these tests.
2. Add 20 drops of the solution of NaCl to a clean, correctly marked test tube.

3. Repeat Step 2 with each of your solutions.
4. Add 5 drops of the solution of $AgNO_3$ to each test tube. Record your observations.
5. The test in this step applies only to those test tubes that contain precipitates. Add 10 drops of the solution of HNO_3 to each of these test tubes, and shake gently. Test a drop of each solution with blue litmus paper. If the paper does not turn pink, add drops of the solution of HNO_3 to the solution in the test tube, with stirring, until it does. Did the precipitates dissolve? Record your observations.
6. Discard the solutions in the test tubes. Wash and rinse the test tubes using distilled water.

Testing with thymol blue

1. Use fresh solutions for these tests.
2. Add 20 drops of the solution of NaCl to a clean, correctly marked test tube.
3. Repeat Step 2 with each of your solutions.
4. Add 5 drops of the solution of thymol blue to each test tube. Shake each test tube gently before recording your observations.
5. Discard the solutions in the test tubes. Wash and rinse the test tubes using distilled water.

Identifying the unknown compound

1. With one possible exception, the characteristic reactions of the unknown compound should be identical to those of one of the known samples. If your unknown is Na_2CO_3, its behavior in solution when tested with $AgNO_3$ may differ slightly from that of a known sample. Differences in the concentrations of these solutions can lead to reactions that result in slightly different colors. Keeping this possible difference in mind, you will be able to identify your unknown compound by matching its reactions with those of one of the known samples.
2. If ambiguities occur, repeat as many of the tests as you find necessary.

Identification of an Unknown Compound

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

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

Section: .....

Instructor: .....

Prelaboratory assignment

1. Match the name of each compound with its formula.

sodium hydrogen phosphate	NaI
ammonia	HNO_3
sodium carbonate	NaCl
sodium sulfate	Na_2CO_3
nitric acid	Na_2HPO_4
sodium chloride	Na_2SO_4
sodium iodide	NH_3

2. List the signals for chemical reactions that you will find in this experiment.

3. What special safety precautions must be observed during this experiment?

Identification of an Unknown Compound

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Results

	HNO_3	$Ba(NO_3)_2$	$Ba(NO_3)_2$ + HNO_3	$AgNO_3$	$AgNO_3$ + HNO_3	Thymol Blue
NaCl						
NaI						
Na_2CO_3						
Na_2HPO_4						
Na_2SO_4						
Unknown No. _____						

My unknown is .....

Questions

1. When an unknown sample that may contain one of the five compounds from this experiment is treated with a solution of $AgNO_3$, a yellow precipitate forms.

   a. Using your record of observations, identify the unknown if possible. Note, however, that your record may show that more than one of the compounds is implicated. Explain your answer carefully.

   b. If more than one compound is implicated, how could you distinguish among them using the tests in this experiment?

2. An unknown sample contains at least two of the five compounds from this experiment. Use the following observations to identify the components of the mixture. Explain your reasoning.

   a. No gas is evolved when the solid is treated with HNO_3. The solution that results is colorless.

   b. A white precipitate forms after the addition of NH_3 and $Ba(NO_3)_2$ to the solution of the unknown. The precipitate does not dissolve after the addition of HNO_3.

   c. A white precipitate forms after the addition of $AgNO_3$ to a solution of the unknown. The precipitate does not dissolve after the addition of HNO_3.

   d. A yellow color results when thymol blue is added to a solution of the unknown.