1. The Identity of an Insoluble Precipitate

Introduction

The properties of any substance depend in part on the chemical bonds that hold the atoms of the substance together. The consequences of this dependence are very important in chemical reactions. Because bonds are formed or broken during a reaction, the properties of product molecules differ from those of reactant molecules. If there is a significant difference, a distinct signal that a chemical reaction has occurred can usually be observed.

One easily seen signal of a chemical reaction is the formation of an insoluble precipitate. This experiment deals with a quantitative interpretation of a reaction in which this signal has appeared.

Purpose

In this experiment, you will examine the reaction between $Ba(NO_3)_2$ and NH_2SO_3H (sulfamic acid) in a hot solution. The identity of the insoluble substance that results from the reaction will be determined from mass relationships.

Concept of the experiment

Known quantities of $Ba(NO_3)_2$ and NH_2SO_3H will be allowed to react in boiling water. Certain covalent bonds in the molecules of sulfamic acid will break slowly during this reaction, and a polyatomic anion will be formed. This anion will combine with the Ba^{2+} cations from $Ba(NO_3)_2$ to form an ionic substance that appears as a white precipitate.

All of this precipitate must be separated by gravity filtration because its mass must be measured. Make sure that you read about this type of filtration in the Introduction to this manual. You will also have an opportunity to practice during the experiment. Take full advantage of this opportunity!

Because you will need to know the mass of the precipitate to determine its identity, you must remove all of the residual water by drying. You will know when the precipitate is dry because its mass will reach a constant value within acceptable error (± 0.002 g, or any other range given to you by your laboratory instructor).

The precipitate will be one of three possible compounds:

1. $Ba(NH_2SO_3)_2$ (barium sulfamate)
2. $BaSO_4$ (barium sulfate)
3. $Ba(NH_2)_2$ (barium amide)

You will determine which one it is from the masses of the precipitate and the limiting reactant (Ebbing/Gammon, Section 3.8).

Because of lack of time, you will be unable to do the experiment more than once. Be careful; avoid major mistakes and systematic errors. Relatively small random errors can be tolerated, however, because you will pool your results with those obtained by other students. As a result, you will have enough data to determine the precision by the method shown in Appendix: Mistakes, Errors,

Accuracy, and Precision. You may do the calculations by hand or, if your laboratory instructor wishes, you can use a computer and the Internet.

Procedure

Getting started

1. Make sure that you have read about gravity filtration in the Introduction to this manual.
2. If you do not understand how to fold the filter paper, ask your laboratory instructor for help.
3. Obtain directions for discarding the solution that you will use in this experiment.

Initiating the reaction

1. You will need about 1.3–1.4 g of $Ba(NO_3)_2$ and about 2.4–2.5 g of NH_2SO_3H. One of these substances is the limiting reactant, as you showed in the Prelaboratory Assignment. You should measure its mass with your most precise balance. You can measure the mass of the other substance on a platform balance, triple beam balance, or an electronic top-loading balance. Your laboratory instructor will give you instructions for using these balances. Use weighing paper in all cases. Record each mass that you measure.
2. Transfer both samples to a 250-mL beaker and add 150 mL of distilled water from a graduated cylinder.

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

3. Using the rubber end of a glass stirring rod equipped with a rubber policeman, stir the mixture until most of the solids have dissolved. The remainder will dissolve when the solution is heated. Do not remove the stirring rod from the beaker.
4. Mark the volume of the solution in the beaker with a marking pencil so that a constant volume can be maintained throughout the course of the reaction.
5. Set up a ring stand with an iron ring, and put a piece of wire gauze on the ring. Adjust the height of the ring so that the wire gauze will be in the hottest part of the flame from a laboratory burner. Do not light the burner until you have made this adjustment.

   CAUTION: Avoid burning your fingers. Do not touch the iron ring or the wire gauze at any time while the solution is being heated.

6. Place the beaker on the wire gauze and heat the solution to a *gentle* boil. Allow the solution to boil for about 40 min after the first cloudiness appears.
7. Stir the solution occasionally without removing the stirring rod. Add increments of distilled water to maintain the original volume.
8. While the solution is boiling, practice filtration.

Practicing filtration

1. Obtain about 0.5 g of powdered $CaCO_3$ (calcium carbonate) on a piece of weighing paper, using a platform balance or a triple beam balance.
2. Transfer the sample to a large beaker. Add 150 mL of distilled water. Stir the mixture thoroughly with the rubber end of another glass stirring rod equipped with a rubber policeman.
3. Filter the mixture using gravity filtration, a technique described in the Introduction to this manual. No trace of $CaCO_3$ should remain in the beaker after filtration is complete.
4. If you wish, repeat with another sample of $CaCO_3$ in water.

Finishing the experiment

1. When the period of heating is completed, remove the flame and cool the beaker to room temperature using cold water or ice.
2. While the beaker and its contents are cooling, obtain the mass of a piece of filter paper using your most precise balance. You will use this filter paper to separate the precipitate from the solution.
3. Filter the cooled mixture containing the precipitate, using the technique that you have practiced.
4. Use a metal spatula to loosen the edge of the filter paper from the filter funnel.
5. Carefully transfer the filter paper and its contents to a beaker that has been labeled with your name. The paper should be upright and never upside down.
6. Place the beaker in an oven at 85°C and allow it to remain there for at least 1 h.
7. During this time, you can review your conclusions about the limiting reactant (see Prelaboratory Assignment). You can also calculate the theoretical yields (Ebbing/Gammon, Section 3.8) of $Ba(NH_2SO_3)_2$, $BaSO_4$, and $Ba(NH_2)_2$ (see Question 2a).
8. After 1 h, remove the beaker from the oven, using tongs, and allow it to cool. Obtain and record the mass of the filter paper and its contents, using your most precise balance.
9. Return the paper to the beaker and the beaker to the oven for an additional 5 min. Remove the beaker, cool the paper, and obtain its mass once again. Continue until a constant mass (± 0.002 g, or any other range stipulated by your laboratory instructor) is reached.
10. Calculate the value of the ratio

    Mass of precipitate/mass of limiting reactant

    and share this information with seven other students. Record their ratios and compare them with your own.

CAUTION: Before you leave the laboratory, make sure that your gas outlet and those of your neighbors are closed.

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

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

Section: .....

Instructor: .....

Prelaboratory assignment

1. The following reactions, shown in unbalanced equations, are pertinent to this experiment. Balance the equations.

   a. ..... $Ba(NO_3)_2$ + ... NH_2SO_3H + ... $H_2O \rightarrow$ .....

   $Ba(NH_2SO_3)_2$ + ... HNO_3

   b. ..... $Ba(NO_3)_2$ + ... NH_2SO_3H + ... $H_2O \rightarrow$ .....

   $BaSO_4$ + ... NH_4NO_3 + ... HNO_3

   c. ..... $Ba(NO_3)_2$ + ... NH_2SO_3H + ... $H_2O \rightarrow$ .....

   $Ba(NH_2)_2$ + ... H_2SO_4 + ... HNO_3

2. When a solution containing 1.4 g of $Ba(NO_3)_2$ and 2.4 g of NH_2SO_3H is boiled, a precipitate forms. This precipitate may be $Ba(NH_2SO_3)_2$, $BaSO_4$, or $Ba(NH_2)_2$. You can find these products in the reactions whose equations you balanced in the first question.

   a. Calculate the number of moles of each reactant.

b. Determine the limiting reactant in each of the following three reactions. The limiting reactant that you determine here will be used directly in this experiment.

   Reaction producing $Ba(NH_2SO_3)_2$:

   Reaction producing $BaSO_4$:

   Reaction producing $Ba(NH_2)_2$:

c. What conclusion can you reach about the limiting reactant in this experiment?

3. What safety precautions are cited in this experiment?

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Results

Mass of paper and $Ba(NO_3)_2$ (g): .....

Mass of paper (g): .....

Mass of $Ba(NO_3)_2$ (g): .....

Mass of paper and NH_2SO_3H (g): .....

Mass of paper (g): .....

Mass of NH_2SO_3H (g): .....

Mass of paper and precipitate (g): .....

.....

.....

.....

Mass of paper (g): .....

Mass of precipitate (g): .....

Ratios (mass of precipitate/mass of limiting reactant) (Include your own result.)

Name of Student	**Ratio**
.....	.....
.....	.....
.....	.....
.....	.....
.....	.....
.....	.....
.....	.....
.....	.....

Questions

1. a. Compute the mean value of the ratio

Mass of precipitate/mass of limiting reactant

and its standard deviation (Appendix: Mistakes, Errors, Accuracy, and Precision), using the shared data, either by hand or, if your laboratory instructor wishes, using a computer and the Internet.

b. Give a mean value of the ratio that is consistent with the precision of the data.

c. Calculate a mass for the precipitate that is consistent with the precision of the data. Use the mean value of the ratio that you calculated in Question 1b and the mass of your limiting reactant.

Student name: ..... Course/Section: ..... Date: .....

2. a. Calculate the expected masses of the three possible products. Use the mass of the limiting reactant and the equations that you balanced in the Prelaboratory Assignment.

$Ba(NH_2SO_3)_2$:

$BaSO_4$:

$Ba(NH_2)_2$:

b. Compare the mass from Question 1c with the masses obtained in Question 2a. Based on this comparison, what is the identity of the precipitate?

3. a. What is the electron-dot formula for NH_2SO_3H? The first two hydrogen atoms are bound to the nitrogen atom, whereas the last hydrogen atom is bound to an oxygen atom. Bonds between one oxygen atom and another do not occur in this compound.

b. What is the electron-dot formula for the anion in the precipitate?

c. What covalent bonds in the sulfamic acid and water molecules must have been broken to form this anion? What bonds, if any, must have formed?