4B

Determining the Empirical Formula of a Compound

A compound you will recall is a substance composed of two or more elements that have been chemically united. In this experiment you will form an iron-oxygen compound by chemically uniting the element iron with the element oxygen. You will use steel wool, which is primarily iron, and react it with the oxygen in the air. (Remember that air is about 20% oxygen.)

The reaction of iron with oxygen is very common; you are probably familiar with this process in the form of rusting. Rusting is a slow process and, under normal lab conditions, the steel wool might take several years to rust. Therefore, to enhance the rusting process, you will dip the steel wool in an ammonium chloride solution, which creates conditions that speed up the reaction.

You will observe that, even with the addition of ammonium chloride, the rusting process still takes time. The experiment cannot be completed in one lab period – in fact, you may not be able to complete the experiment for a week or so. Some forethought is necessary on your part so that you can plan your activities. By the end of the experiment (Part III) you will have collected data that will enable you to determine the empirical formula of the compound which has formed.

In most cases in daily life, the rusting process is considered undesirable and billions of dollars are spent on rust prevention. Chemists regularly conduct experiments such as this one in their search for improved methods of slowing rust formation. Such experimenting has lead to the development of new protective coating materials for iron as well as more sophisticated techniques such as cathodic protection. Cathodic protection is a technique that prevents the iron atoms from transferring their electrons to oxygen, thus preventing the rust reactions from occurring.

OBJECTIVES

1. to form a compound from the elements iron and oxygen
2. to determine the empirical formula of the compound produced in this chemical reaction

SUPPLIES

Equipment

ring stand
ring support
lab burner
crucible
crucible tongs
water-soluble marker
pipestem triangle
heat resistant mat
centigram balance
lab apron
safety goggles
medicine dropper

Chemical Reagents

steel wool, fine
1*M* ammonium chloride solution, NH_4Cl

PROCEDURE

CAUTION

Ammonium chloride solution is mildly corrosive. Keep it off your skin and clothing and out of your eyes. Wash away any spills with plenty of water.

Part I: (First Day)

1. Put on your lab apron and safety goggles.
2. Obtain a clean, dry crucible (without a lid) and accurately determine its mass. Enter this value in your copy of Table 1.
3. Place a clump of steel wool on the balance pan and add or remove steel wool until you have 1.50 g.
4. Take the 1.50 g of steel wool and place it loosely (allowing air into it) into crucible. Reweigh the crucible and contents, and enter the relevant information in your data table.
5. Take the crucible and contents to the "dunking station" that your instructor has set up. Here you will find the ammonium chloride solution in a large beaker.
6. Remove the ball of steel wool from the crucible and, using crucible tongs, gently submerge the steel wool in the ammonium chloride solution. Wait until the steel wool is thoroughly soaked, then remove it and carefully squeeze out any excess solution.
7. Replace the moistened steel wool in the crucible. Label the crucible with a water soluble marker; indicating your name and class.
8. Place your crucible, uncovered, in the designated storage location. Now the waiting begins!

CAUTION

Objects that have been heated may appear to have cooled when, in fact, they are still hot. Serious burns can result.

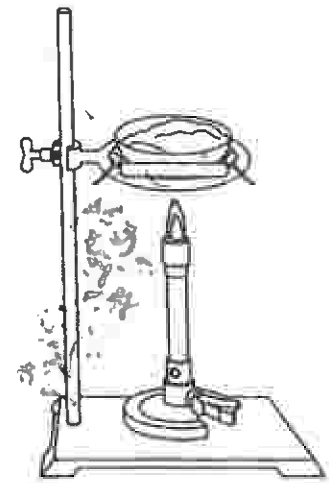
Part II: (Next Several Days)

1. Over the next week, check your crucible each day to observe any changes.
2. Each day, use a medicine dropper to add a few drops of ammonium chloride solution so that the steel wool is remoistened.
3. Continue these activities for as long as your instructor advises. The time required usually varies between four and seven days. Since Part II requires only a few minutes during each class, you can expect to carry on with activities other than this experiment during the waiting period.

Part III: (Final Day)

1. Retrieve your crucible from the storage location.
2. Set the crucible in a heating apparatus, as shown in Figure 4B-1. The crucible will sit inside a pipestem triangle which is connected to a ring support.
3. Heat the crucible and contents with the lab burner adjusted to a high temperature. The outside of the crucible will glow a dull red when it becomes very hot. Continue to heat the crucible at red heat for at least ten more minutes, and then turn off the burner.
4. Let the crucible cool so that you can touch it. CAUTION: The ring stand and accessories can burn you even after several minutes. If you are in doubt about the ring stand, you can test how hot it is by touching it with a piece of wet paper towel.
5. Determine the mass of the crucible and contents (a compound) and record this in your data table.
6. Clean up your materials according to the reagent disposal instructions and wash out and dry the crucible.
7. Before you leave the laboratory, wash your hands thoroughly with soap and water.

Figure 4B-1 *Apparatus for heating the steel wool on the final day*



REAGENT DISPOSAL

The ammonium chloride solution can be rinsed down the sink with large amounts of water. The crucible's contents can be placed in a garbage can.

POST LAB CONSIDERATIONS

In order to find the empirical formula of the compound produced, you need to know how many moles of iron and oxygen atoms reacted. Since you know the mass of iron reacted (you must assume here that all 3.0 g reacted), you can convert "mass of iron" into "moles of iron." The mass of oxygen reacted can be determined by examining Table 1 and applying some common sense: mass of compound – mass of iron = mass of oxygen. Now, the mass of oxygen atoms can be converted to moles of oxygen atoms. (Note: The atomic mass of O is 16.0, not 32.0.)

The next step towards determining the empirical formula is to calculate the ratio

$$\frac{\text{moles of O atoms}}{\text{moles of Fe atoms}}$$

Finally, if this mole ratio contains decimals, convert it into a whole number ratio. An example is given in the chart which follows.

	Moles	Mole Ratio	Ratio Doubled	Ratio Tripled
Element X	2.42	1	2	3
Element Y	6.46	2.67	5.33	8.01

You double, triple, etc., the mole ratio until you end up with a value very close to a whole number. In the example being discussed, the whole number ratio is 3:8. Hence, the empirical formula of the compound would be X_3Y_8.

EXPERIMENTAL RESULTS

Table 1

Before the Reaction	
Mass of crucible + iron (steel wool)	
Mass of crucible	COMPLETE IN YOUR NOTEBOOK
Mass of iron	
After the Reaction	
Mass of crucible + compound	
Mass of crucible	COMPLETE IN YOUR NOTEBOOK
Mass of compound (iron + oxygen)	

ANALYSIS OF RESULTS

1. Calculate the number of moles of iron atoms that reacted. Enter Analysis 1–4 values in a table similar to the one in Post Lab Considerations.
2. Determine the mass of oxygen atoms that reacted.
3. Calculate the number of moles of oxygen atoms that reacted.
4. Calculate the smallest whole-number ratio of oxygen atoms to iron atoms.
5. Write the empirical formula for the compound.
6. Look up the ion charges for iron and oxygen, and predict two possible formulas for iron oxide.
7. How does your experimentally-determined formula compare with your predicted formulas?

FOLLOW-UP QUESTIONS

1. Potassium persulfate is used in photography to remove the last traces of unwanted chemicals from photographic papers and plates. A 0.8162 g sample was found to contain 0.2361 g of potassium, 0.1936 g of sulfur; the rest was oxygen. What is the empirical formula of this compound?

CONCLUSION

State the results of Objective 2.