# Activity 16: The Formation of Iron Sulfide - Pyrrhotite Maine Geological Survey



### **Objectives:**

To introduce students to the concept of the chemical formation of minerals from a hot, semi-liquid melt; to introduce basic stoichiometric concepts regarding a minerals formula (in this case FeS); and to allow students to develop some practice in distinguishing among elements and a compound made from those two elements.

#### Time:

This activity is designed to last two periods.

#### **Background:**

Chemistry and mineralogy have been intertwined as scientific disciplines since about the Bronze Age. The ability to extract specific elements from their naturally occurring minerals, and then combine these elements into new or purer compounds and alloys has been the basis of every technological advance from the low carbon steel of the Roman Empire to the microchips of the 1990's. Pyrrhotite, which is chemically called iron sulfide, has the ideal formula FeS (Fe = iron, S = sulfur). This formula means that in ANY AMOUNT of pyrrhotite, the ratio of iron to sulfur will be 1:1. The equation for the reaction that produces the iron sulfide is:

Fe + S  $\rightarrow$  FeS (heat)

One atom of iron (or a million atoms of iron) and one atom of sulfur (or one million atoms of sulfur) react with heat to make one molecule of iron sulfide (or a million molecules of iron sulfide). The new compound (mineral) has different chemical and physical properties from the elemental iron and sulfur out of which it was formed.

### Materials:

Each group of students will need the following:

- 7 grams of iron filings
- 4 grams of powdered sulfur
- 2 number-two (No.2) solid rubber stoppers
- 2 sheets of massing paper
- 1 magnet
- Three 20x150 mm Pyrex test tubes
- 250 ml beaker
- Fisher burner with attached burner hose
- 10 ml graduated cylinder
- Test tube clamp (hand held)
- Tweezers
- A plastic or metal scoop for transferring dry chemicals
- A test tube brush

Each class as a whole will need:

- 75 ml of 6 molar hydrochloric acid (HCl)
- 75 ml of kerosene
- a burner lighter
- a balance to weigh the iron and sulfur

While none of the chemicals in this activity are particularly hazardous or toxic, disposal of used chemicals should follow the protocol developed by the school. Each student will need pens, notebook, and safety goggles.

### **Procedure:**

I. Have the students weigh out 7 grams of iron filings and 4 grams of powdered sulfur respectively. Place each of these materials on a separate piece of massing paper. Run a magnet UNDER each sheet of paper and record the results. (If time is an issue, you may wish to have the samples of iron and sulfur weighed out and placed on separate pieces of paper prior to the start of class. This reduces the time needed considerably, but precludes student use of the balance.)

**II.** Place a few grains of sulfur in 3 ml of hydrochloric acid; record the results. Discard this material as dictated by the school's chemical handling procedures. Rinse your test tube. Repeat this test with a few iron filings in the HCl, observe and record the results. The amounts of iron and sulfur tested should be about the size of a paper match head.

**III.** Place a few iron filings into 3 ml of kerosene; stopper, shake **GENTLY**, observe and record results. Discard this material into the organic solvents container; rinse the test tube and repeat this test with 3 ml of kerosene and sulfur. Record the results.

**IV.** Mix the remaining iron and sulfur thoroughly together and transfer this mixture to a clean, dry test tube. **THIS NEXT PART MUST BE DONE UNDER A FUME HOOD; THE STUDENTS MUST BE WEARING THEIR SAFETY GOGGLES.** With the fume hood running, and holding the test tube with the test tube clamp, heat the test tube containing the iron and sulfur vigorously. Gases will form at the mouth of the test tube and catch on fire, this is normal; keep on heating. When the bottom of the test tube is red hot and glowing you will need to heat for an additional 30 seconds. After this time remove the tube from the heat and immediately quench this in a 250 ml beaker half full of cold water. The tube will break and gasses will shoot out the open end, invert the tube and quench the mouth end. Remove the beaker from under the fume hood and recover the solid product from the bottom of the beaker. Discard the broken glass as appropriate.

V. Remove the product and examine it; test it with a magnet and record your observations. Place a small match head sized piece of the product into 3 ml of kerosene and record the results. Test a very small piece of the product with 3 ml of hydrochloric acid in a test tube. **DO NOT STOPPER, and PERFORM THIS TEST UNDER THE FUME HOOD.** Use the wafting technique to obtain a faint whiff or smell of the gas being produced. Describe the smell; discard this down the sink under the fume hood with plenty of water. Record your results. The tests performed on the product in this section prove that a new substance, iron pyrrhotite, with new properties has been produced.

### **Special Safety Precautions:**

The heating of the iron and sulfur, as well as the testing of the compound with hydrochloric acid **MUST BE PERFORMED UNDER A FULLY FUNCTIONING FUME HOOD**. If one is not available the TEACHER may elect to produce one sample of the compound by performing the heating outdoors and then have the class test bits of the new compound, again testing it with hydrochloric acid outdoors. If you have any questions on this, obtain some assistance from the chemistry teacher. Students MUST wear their safety goggles while testing, heating and handling chemicals.

**NOTE TO TEACHERS**: There are a number of iron sulfide compounds which can be produced, including pyrite which is  $FeS_2$ , and marcasite which is also  $FeS_2$  but crystallizes in the orthorhombic system. Technically pyrrhotite is a mineral with a variable formula which ranges from  $Fe_5S_6$  to  $Fe_{16}S_{17}$ . The general formula for pyrrhotite is  $Fe_xS_{x+1}$ . This particular activity will most likely produce some mixture of the above three minerals in the test tube. The tube contents will NOT look like the common fool's gold ( $FeS_2$ ) one sees from typical sulfide deposits. Astute students may notice this and question you about it; this can lead to a good discussion of natural as opposed to man-made materials. What is lacking in the lab process that exists in nature?

Summary of Test Results				
Substance	Magnetic?	Tested with HCl	Kerosene	
Iron	Voc	No reaction, or very tiny	No reaction	
	res	bubbles, faint, if any odor		
Sulfur	No	Floats on top	Dissolves	
Iron Sulfide	Weakly magnetic	Very active bubbling, rotten egg	No reaction	
	in small chunks	smell, hydrogen sulfide gas		

### Follow-Up:

See Activity #22 (<u>Covellite or Chalcocite???</u>) for a more subtle variation of this activity.

Have students observe a piece of iron pyrrhotite formed in nature, such as the classic Mexican crystals, and compare and contrast the iron pyrrhotite they made with that formed in nature. How do you account for the differences? Can you prove that natural iron pyrrhotite really is FeS?

Look up the chemical formula for the Maine State gem stone, tourmaline. Have students copy it off the board. Ask them to describe the conditions under which tourmaline probably forms.

## **References:**

Activity developed by Duane L. Leavitt.



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# **Student Sheet**

## Purpose:

To contrast the properties of two elements, iron and sulfur, with a new substance made from those elements. By simulating a natural process, you will demonstrate the chemical formation of an iron sulfide mineral from a hot, semi-liquid melt.

## Materials:

Each group of students will need the following items: 7 grams of iron filings, 4 grams of powdered sulfur, 2 sheets of weighing paper, 3 Pyrex test tubes 20x150 mm, 2 number two solid rubber stoppers, a magnet, a 250 ml beaker, a Fisher burner with attached burner hose, a 10 ml graduated cylinder, a hand held test tube clamp, tweezers, a metal or plastic scoop for transferring chemicals, and a test tube brush.

Each student will need safety goggles, pens, and notebook. The class as a whole will need a balance for weighing out chemicals, a bottle of hydrochloric acid, a bottle of kerosene and a burner lighter. *BE CERTAIN YOU FOLLOW YOUR TEACHER'S INSTRUCTIONS FOR THE HANDLING, USE, AND DISPOSAL OF THE CHEMICALS INVOLVED IN THIS ACTIVITY.* 

### **Procedure:**

Before doing any part of the following activities, put on your safety goggles, and KEEP them on for the duration of the activity. For each step, record all of your observations in the data table.

**PART I. MAGNET TEST.** Place 7 grams of iron filings on a piece of paper; repeat this process for 4 grams of powdered sulfur. Run a magnet under the sulfur; repeat this process with the iron filings; record your observations.

**PART II. ACID TEST.** Take a few grains of iron filings, place them in a Pyrex test tube. Measure out three ml of hydrochloric acid with your graduated cylinder and add this acid to the iron filings in the test tube; rinse your graduated cylinder before further use. Stopper the test tube and shake gently. Remove the stopper and sniff the test tube; observe the test tube and its contents. Record your results. Repeat the above process with the sulfur; observe and record your results. Discard the chemicals as directed by the teacher; clean your test tube before further use.

**PART III. KEROSENE TEST.** Place a few grains of iron filings into a test tube. Measure out 3 ml of kerosene in the graduated cylinder and add this to the iron filings. Stopper, shake GENTLY and observe. Repeat this test for the sulfur. While kerosene has its own distinct smell, you may wish to perform the smell test anyway to see if the smell of the kerosene changes. Discard these materials as directed by the teacher and clean your test tubes before further use.

**PART IV. MAKING IRON SULFIDE.** Thoroughly mix together the remaining sulfur and iron left from Part I above and place them in a clean, dry test tube. The next procedure must be performed **UNDER A FUME HOOD**; your teacher will instruct you in its use.

With the fume hood running, heat the test tube containing the iron and sulfur mixture. You need to have the bottom of the test tube about an inch above the wire mesh at the top of the Fisher burner; this is the hottest part of this particular burner's flame. As you heat the mixture, gases will form at the mouth of the test tube. They may catch on fire and you may see blue flames coming out of the test tube mouth; this is normal; keep heating. When the bottom of the test tubes glows red hot and the contents inside are glowing red, you will need to heat thirty seconds longer. After this time has passed, remove the test tube from the burner flame and immediately quench it in the 250 ml beaker which should be half full of water. The end you have been heating will probably break off in the beaker; invert the test tube and quench the other end. Remove the beaker and the contents from under the hood.

**PART V. TESTING IRON SULFIDE.** Remove the solid chunk of material that you have formed from the beaker of water; dry it off and examine it; record your observations and description of the product. Test a small piece of this material with the magnet and describe the results. Take a small chunk and put it in a test tube with 3 ml of kerosene. Stopper the test tube and shake gently; describe the results, and record.

Take another **VERY SMALL** chunk and place it in a clean, dry, test tube. Add 3 ml of hydrochloric acid. **DO NOT STOPPER OR SHAKE, AND BE CERTAIN TO DO THIS UNDER THE FUME HOOD; MAKE SURE THE HOOD IS RUNNING**. Use the wafting technique to gently draw some of the gas being produced from the reaction between the product and the acid to your nose. Describe the smell, and record.

**PART VI. CLEANUP.** Dispose of all broken glass and remaining chemicals as directed by your teacher. Clean your test tubes, graduated cylinders, and beakers before leaving.

Data Table					
TEST	IRON	SULFUR	NEW COMPOUND		
Color					
Magnetism					
Reaction with HCl					
Reaction with kerosene					
Comments					

### **Questions:**

1. Describe four ways you can distinguish iron from sulfur.

2. How does the product you created look compared to the elements it is composed of? Explain.

3. How can you PROVE that the substance produced in Part IV is a new product, a new chemical substance? BE SPECIFIC in your answer.

4. Does the product look like gold to you? Is it heavy? Explain how it might be confused with gold.