# THE PREPARATION OF THE ELEMENT IODINE

The purpose of this experiment is to prepare crystals of iodine and observe some properties of iodine.

The iodine is prepared by mixing potassium iodide (KI) and manganese (IV) oxide,  $MnO_2$ , with phosphoric acid,  $H_3PO_4$ . The chemical equation for this reaction is

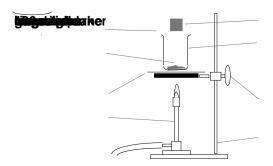
$$6 \text{ KI(s)} + 3 \text{ MnO}_2(s) + 4 \text{ H}_3 \text{PO}_4 \longrightarrow 3 \text{ I}_2(s) + \text{Mn}_3(\text{PO}_4)_2(s) + 2 \text{ K}_3 \text{PO}_4(s) + 6 \text{ H}_2 \text{O(I)}$$
.

### **PROCEDURE**

Important: Your observations must include a visual description of all the chemicals used, in addition to a description of the reactions and changes you observe.

Because of the hazards associated with phosphoric acid and iodine you must wear safety goggles and latex gloves during the experiment.

1. Set up a stand, ring and gauze pad **IN A FUME HOOD**, as shown below. Also, get a 100 mL beaker, a watch glass, a stirring rod, a bunsen burner and a striker. Adjust the height of the ring on the stand so that the ring is about 5 cm (2 inches) above the top of the burner.



- 2. Put a plastic weighing boat on the balance and "zero" the balance. Then weigh **between 0.9g and 1.1g** of **potassium iodide** into the weighing boat.
- 3. Put a second weighing boat on the balance, zero the balance, and weigh **between 0.5 g and 0.7 g** of **manganese (IV) oxide** into the weighing boat.
- 4. Pour both the potassium iodide and manganese (IV) oxide into a 100 mL beaker and stir the solid chemicals together for a few seconds.
- 5. Use a dropping bottle to place 1 mL of concentrated phosphoric acid into a 10 mL graduated cylinder. Carefully pour the phosphoric acid into the beaker containing the chemicals, letting the acid drain into the beaker for 15 seconds to make sure that none of the acid dribbles down the outside of the graduated cylinder. Carefully wash out the graduated cylinder several times using a slow flow of water from the tap (so as not to blast acid into your face).
- 6. Place the beaker onto the gauze pad on the ring stand, place a watch glass over the beaker and place an ice cube on the watch glass.
- 7. Light the burner and make sure the flame is directly under the beaker. Use a flame with a low heat; that is, a small bushy flame with the air vent closed. **Record your observations as the reaction occurs.** Turn off your bunsen burner when the purple gas in the beaker has noticeably decreased in amount.
- 8. After turning off the heat, allow the beaker and watch glass to cool for 5 minutes. Then, carefully remove the ice cube, pour off any water which has accumulated in the watch glass, turn the watch glass over and **describe the crystals** which have formed on the underside of the watch glass.

### THE PROPERTIES OF IODINE CRYSTALS

- 1. Scrape a few crystals of iodine (an amount that would form a little pile the size of this "o") into each of 3 small test tubes. **IMPORTANT: DO NOT USE TOO MUCH!**
- 2. Into the first test tube add about **3 cm height of water**, stopper and shake the contents for about 15 seconds. Record the colour of the water after shaking and estimate the amount of iodine that has dissolved (for example, very little, about half, most, all).
- 3. Into the second test tube add about 3 cm height of hexane, stopper and shake the contents for about 15 seconds. Record the colour of the hexane before adding it to the test tube and after shaking with iodine. Estimate the amount of iodine that has dissolved.
- 4. Take the 3rd test tube to the fume hood, place the tube in a test tube holder, as demonstrated by your teacher, and hold the bottom of the tube at an angle over a flame for about a minute. Be careful not to let the metal test tube holder get in the flame the heat will melt your latex glove and cause a painful burn. **Record your observations**.

### **QUESTIONS**

- 1. In which liquid is iodine more soluble: water or hexane? Explain your answer.
- 2. A student puts a crystal of iodine on a watch glass at room temperature and leaves the watch glass and crystal overnight. The next morning, the crystal has disappeared. Suggest a possible reason why the crystal disappears. (You are entitled to make a guess. No, the crystal is not taken by someone.)
- 3. You have a mixture of sand and iodine. Suggest a way to separate and remove the iodine from the sand. (There is more than one way to do this, so any reasonable method is acceptable. No, you can't use a pair of tweezers and a microscope because it will take until you are 600 years old to finish the job.)

## **CONCLUSIONS**

Summarize the properties and behaviour of iodine. In other words, describe everything you learned about iodine in this experiment?