Mystery Powder from a Crime Scene

A white powder is collected at a crime scene. Your job as a forensic chemist is to determine the chemical properties of the powder and attempt to identify it. You will measure the pH, solubility in terms of conductivity, and melting point of the powder. You will also observe the reaction with iodine solution, and an acidified ferric ion solution. By comparing the properties of the crime scene powder to those of known substances you will report what you think is the identity of the powder.

OBJECTIVES

- Measure the pH of solutions made from powdered substances
- Measure the conductivity of solutions made from powdered substances
- Observe the chemical reactions with iodine solution, and acidified ferric ion solution
- Measure the melting points of powders
- Report the identity of an unknown powder based on physical and chemical properties

MATERIALS

computer or Chromebook
Graphical Analysis 4 app
Vernier data-collection interface
pH Sensor
Conductivity Probe
Vernier Melt Station
mortar and pestle
50 mL graduated cylinder
6 test tubes, 13 × 100 mm
test tube rack
goggles
3 plastic droppers
stirring rod
balance with resolution to 0.1 g
baking powder
sodium bicarbonate, (baking soda)
aspirin
sucrose
cornstarch
iodine solution
0.2 M $Fe(NO_3)_3$ solution in HNO_3

PROCEDURE

Caution: *Obtain and wear goggles during this experiment. Be careful not to ingest any solution or spill any on your skin. Inform your teacher immediately in the event of an accident.*

Part I Chemical and Physical Properties

1. Obtain and wear goggles.
2. Prepare a table similar to the one in the Evidence Record to record the results of your testing.
3. Dissolve about 0.1 g of each known powder in 15 mL of distilled water in separate, labeled test tubes. Stir with a clean stirring rod.
4. Dissolve about 0.1 g of powder from the crime scene in 15 mL distilled water in a 6th test tube. Label the test tube.
5. If your Conductivity Probe has a switch, set it to 20,000 µS/cm. Connect the Conductivity Probe to the data-collection interface, and then connect the interface to your Chromebook, computer or mobile device. Launch Graphical Analysis.
6. Measure the conductivity of each solution and record the value in the Evidence Record. Rinse the Conductivity Probe with distilled water between measurements.
7. Close Graphical Analysis 4 and disconnect the Conductivity Probe.
8. Connect the pH Sensor to the data-collection interface, and then connect the interface to your Chromebook, computer, or mobile device. Launch Graphical Analysis.
9. Measure the pH of each solution and record the values in the Evidence Record. Rinse the sensor in distilled water between measurements.
10. Add 5 drops of iodine solution to each test tube. Stir and observe. Record your observations in the Evidence Record. **WARNING**: *Iodine solution: Skin and serious eye damage, corrosion or irritation*.
11. Dispose of the waste as directed by your instructor. Rinse all the test tubes thoroughly.
12. Place about 0.1 g of each powder in the test tubes again.
13. Add about 15 mL of distilled water to each test tube and stir with a clean stirring rod.
14. Add 10 drops of $Fe(NO_3)_3$ solution to each test tube. Carefully observe each test tube. Note that the $Fe(NO_3)_3$ solution is prepared with 1.0 M HNO_3. **WARNING**: *Acidified iron(III) nitrate solution,* $Fe(NO_3)_3$: *Skin and serious eye damage, corrosion or irritation*.
15. Record your observations in the Evidence Record.
16. Dispose of the waste as directed by your instructor. Rinse all the test tubes thoroughly.

Part II Determine the Melting Point

17. Prepare a sample for melting.
    a. Pack a capillary tube 3–4 mm (~1/8 inch) deep with a crime scene sample by inserting the open end into a small pile of the solid. A small amount of the solid will be pushed up into the tube.
    b. Wipe off any loose solid that is on the outside of the capillary tube.
    c. Tap the closed end of the capillary tube on the desk top to compress the sample into the closed end.
    d. (optional) To further pack down the sample in the tube, drop the capillary tube (closed end down) down a section of glass tubing that has been set up for this purpose.
    e. Carefully insert the capillary tube of solid into one of the three slots in the heating block of the Melt Station.
    f. Rotate the Melt Station up or down slightly to get the best view of the solid sample through the viewing lens.

18. Connect the Vernier Melt Station to your Chromebook, computer, or mobile device. Launch Graphical Analysis 4.

19. You are now set up to take temperature data for up to 20 minutes.

20. In the first trial, you will want to observe the melting process and make a rough estimate of the melting temperature of your sample. Do not worry if the heating rate is a bit too rapid, and the sample melts too quickly. To do this:
    a. Start data collection.
    b. On the Melt Station, turn the control knob to a setting of 180ºC. The red light will turn on indicating active heating.
    c. Carefully observe your sample. When the solid begins to melt, click or tap on the graph and note this initial melting temperature in your Evidence Record. When the entire solid has completely melted, click or tap on the graph again and note the final melting temperature in your Evidence Record. The two values describe the estimated melting temperature range of your substance.
    d. If the solid does not melt by the time the temperature gets to 150ºC, turn the control knob to the 220ºC setting. Continue observing your sample, and if the sample begins to melt, mark the temperatures on the graph as previously described.
    e. If the sample has not melted by the time the temperature gets to 190ºC, turn the knob to the Rapid Heat setting. When the sample finally begins to melt, note the temperatures from the graph as previously indicated.
    f. When you have determined the approximate melting temperature range for the sample, stop data collection. Discard the capillary tube and sample as directed by your instructor.
    g. On the Melt Station, turn the control knob to the Fan/Cooling setting to get ready for the next trial. The blue light will turn on indicating that the fan is cooling the Melt Station.

21. Now that you have a rough idea of the melting temperature, a more accurate determination can be made. Prepare a new sample in a capillary tube, as described in Step 17, to determine the melting temperature.
    a. Start data collection.
    b. On the Melt Station, turn the control knob to the Rapid Heat setting.
    c. Carefully observe the temperature vs. time graph. When the temperature is within approximately 10ºC of the lowest possible melting temperature of your sample, turn the control knob to a temperature setting corresponding to your expected melting temperature.
    d. Carefully observe your sample. When the solid begins to melt, click or tap on the graph and record the initial melting temperature in your Evidence Record. When the entire solid has completely melted, tap again and record this as the final melting temperature. The two values recorded describe the melting temperature range of your substance. When you are finished with this step, stop data collection.
    e. Discard the capillary tube and sample as directed by your instructor.
    f. On the Melt Station, turn the control knob to the Fan/Cooling setting to get ready for the next trial.

22. Repeat this process for each known powder.

23. At the end of the experiment, record the melting temperature range of the second run for each substance.

24. Turn the control knob on the Melt Station to Off.

EVIDENCE RECORD

Part I Chemical and Physical Properties

Powder	Conductivity of solution (µs/cm)	pH of solution	Observations with iodine solution	Observations after addition of Fe^{3+}
Crime scene				
Baking powder				
Baking soda				
Aspirin				
Cornstarch				
Sucrose			;	

Part II Melting Data

Powder	Rough initial melting temp (°C)	Rough final melting temp (°C)	Second initial melting temp (°C)	Second final melting temp (°C)	Range of melting temps (°C)
Crime scene					
Baking powder					
Baking soda					
Aspirin					
Cornstarch					
Sucrose					

CASE ANALYSIS

1. From the Chemical and Physical evidence, what did you learn about the powder from the crime scene. Make sure to include enough detail to support your conclusion.

2. Why was it important to note that the ferric nitrate solution was made in nitric acid? How does this affect your observations and conclusions?

3. From the Melting Point data, what did you learn about the powder from the crime scene? Make sure to include enough detail to support your conclusion.

4. What additional tests on the powder could also be performed?

CASE REPORT

When you write your case report, make sure to include the following:

- How you determined the chemical and physical properties of the powders.
- How you determined the melting temperature of the powders.
- How you used the data to determine the identity of the crime scene powder.
- Graphs and supporting data that you collected during the experiment.