

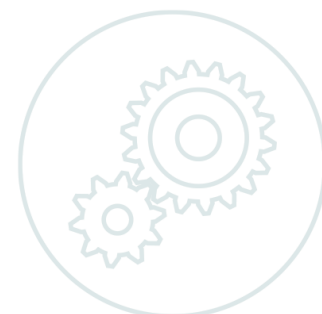
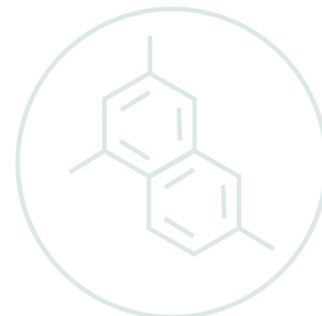
NSTA National 2022 Houston, TX

Forensic Chemistry

Experiments:

Mystery Powder from a Crime Scene

- Go Direct pH
- Go Direct Conductivity
- Go Direct Melt Station

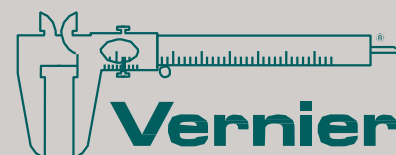


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Mystery Powder from a Crime Scene

A white powder is processed 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 hydrochloric acid and ferric ions. 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

1. Measure the pH of solutions made from powdered substances.
2. Measure the conductivity of solutions made from powdered substances
3. Observe the chemical reactions with hydrochloric acid, iodine solution and ferric ion
4. Measure the melting points of powders
5. Report the identity of an unknown powder based on physical and chemical properties

MATERIALS

Chromebook, computer, **or** mobile device

Graphical Analysis 4 app

Go Direct pH Sensor

Go Direct Conductivity Probe

Go Direct Melt Station

Electrode Support

balance

ring stand

6 test tubes, 13 x 100 mm

test tube rack

goggles

3 plastic droppers

stirring rod

chemical scoop or spatula

baking powder

sodium bicarbonate

aspirin

sucrose

cornstarch

iodine solution

0.2 M $\text{Fe}(\text{NO}_3)_3$ solution in HNO_3

PRELAB

1. Identify the ranges of pH values expected when testing acids, bases and neutral solutions.
2. The conductivity probe measures the electrical conductivity of a solution. Ionic compounds conduct electricity better in solution than molecular substances. Write dissociation equations for 1) sodium bicarbonate and 2) sucrose in water.

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. Prepare a table similar to the one in the Evidence Record to record the results of your testing.
2. 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.
3. Dissolve about 0.1 g of powder from the crime scene in a 6th test tube. Label the test tube.
4. Connect the Conductivity Probe to your Chromebook, computer, or mobile device. Launch Graphical Analysis 4.
5. Set up the data-collection mode.
 - a. Click or tap Mode to open Data Collection Settings. Change Mode to Event Based/Selected Events.
 - b. Enter **Sample** as the Event Name. Leave Units blank. Click or tap Done.
6. Click or tap Collect. Measure the conductivity of each solution and click or tap KEEP to record the value in the data table. Rinse the conductivity probe with distilled water between measurements. Keep track of the sample number in the Graphical Analysis data table and substance tested.
7. Save the conductivity data as directed.
8. Close Graphical Analysis 4 and Disconnect the Conductivity Probe.
9. Connect the pH Probe to your Chromebook, computer, or mobile device. Launch Graphical Analysis 4.
10. Set up the data-collection mode.
 - c. Click or tap Mode to open Data Collection Settings. Change Mode to Event Based/Selected Events.
 - d. Enter **Sample** as the Event Name. Leave Units blank. Click or tap Done.

11. Click or tap Collect. Measure the pH of each solution and click or tap KEEP to record the value in the data table. Record the pH values in the same order as before. Rinse the pH probe with distilled water between measurements.
12. Save the pH data as directed.
13. Add 5 drops of iodine solution to each test tube. Stir and observe. Record your observations in the Evidence Record.
14. Dispose of the waste as directed by your instructor. Rinse all the test tubes thoroughly.
15. Place about 0.1 g of each powder in the test tubes again.
16. Add about 15 mL of distilled water to each test tube and stir with a clean stirring rod.
17. Add 10 drops of $\text{Fe}(\text{NO}_3)_3$ solution to each test tube. Carefully observe each test tube. Note that the $\text{Fe}(\text{NO}_3)_3$ solution is prepared with 1.0 M HNO_3 .
18. Record your observations in the Evidence record.
19. Dispose of the waste as directed by your instructor. Rinse all the test tubes thoroughly.

Part II Determine the Melting Point

1. 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 desktop 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.
2. Connect the Melt Station to your Chromebook, computer, or mobile device. Launch Graphical Analysis 4.
3. Start the data-collection program, and then choose New from the File menu. You are now set up to take melting temperature data for up to 20 minutes.

4. 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 180°C. The red light will turn on indicating active heating.
 - c. Carefully observe your sample. When the solid begins to melt, tap on the graph and note this initial melting temperature in your Evidence Record. When the entire solid has completely melted, tap 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 150°C, turn the control knob to the 220°C 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 190°C, turn the knob to the Rapid Heat setting. When the sample finally begins to melt, mark 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.
15. 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 13, 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 10°C 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, 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.
16. Repeat this process for each known powder.

17. Save the data file by selecting Save from the file menu.
18. At the end of the experiment, record the melting temperature range and turn the control knob on the Melt Station to Off.

EVIDENCE RECORD

Part I Chemical and Physical Properties

Powder	Sample Number in Graphical Analysis	Conductivity of solution ($\mu\text{S}/\text{cm}$)	pH of solution	Reaction with iodine solution	Reaction with Fe^{3+}	Color of solutions after addition of Fe^{3+}
Crime scene						
Baking powder						
Baking soda						
Aspirin						
Cornstarch						
Sucrose						

Part II Melting Data

Powder	Rough initial melting temp ($^{\circ}\text{C}$)	Rough final melting temp ($^{\circ}\text{C}$)	Second initial melting temp ($^{\circ}\text{C}$)	Second final melting temp ($^{\circ}\text{C}$)	Range of melting temps ($^{\circ}\text{C}$)
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.