Forensic Chemistry: Poisoned Wine Mystery Powder

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HANDS-ON ACTIVITIES

The Case of the Poisoned Wine

- Go Direct SpectroVis Plus

Mystery Powder from a Crime Scene

- Go Direct pH
- Go Direct Conductivity

The Case of the Poisoned Wine

Detectives were called to the scene of a dinner party at 321 Main Street in Riddle, Oregon, after some guests at a dinner party died from what is suspected as poisoned wine. Other guests were taken to the hospital. Some died, some are in grave health, and the condition of others is improving. In one case, the crime scene technicians recovered 73 mL of wine from the glass used by one of the victims who died. This sample has been tagged and submitted to the lab.

Why did some guests die and others not?

Your job is to determine what, if anything, was used to poison the wine and why some guests died while others didn't.

OBJECTIVES

- Use spectroscopy to identify an unknown poison.
- Use Beer's law to determine the concentration of the poison.
- Learn the importance of carefully prepared standards.
- Prepare a detailed report of your findings.

MATERIALS

Chromebook, computer, or mobile device
Spectral Analysis app
Go Direct SpectroVis Plus
7 cuvettes and lids
distilled water
waste beaker
droppers
6 test tubes
test tube rack
goggles
stirring rod
two 10 mL pipettes or graduated cylinders
lint-free tissues
fresh, uncontaminated wine sample
evidence wine sample from crime scene
known poison solutions (simulated):

    Prussian blue
    PCB-11 (yellow)
    Schloss green

PROCEDURE

Part I Identify the Poison

1. 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.

2. Prepare a blank by filling a cuvette 3/4 full with distilled water.

3. Launch Spectral Analysis. Connect the Go Direct SpectroVis Plus to your Chromebook, computer, or mobile device. Click or tap Absorbance *vs.* Wavelength.

4. To calibrate the spectrometer, place the blank cuvette in the spectrometer taking care to align the cuvette correctly, and select Finish Calibration. **Note**: If necessary, wait for the spectrometer to warm up before selecting Finish Calibration.

5. Prepare a test sample by filling a cuvette 3/4 full with fresh wine.

6. Collect spectrum data for fresh wine.
   a. Remove the blank cuvette, and place a cuvette of fresh wine into the cuvette slot. Make sure to align the cuvette correctly.
   b. Click or tap Collect. The absorbance *vs.* wavelength spectrum will be displayed. Click or tap Stop.
   c. To rename the run, click or tap More Options next to Data Set 1 on the table, then select Rename Data Set.
   d. Enter **Fresh Wine** for the name of this run. Click or tap RENAME.

7. Prepare a sample of wine from the crime scene by filling a cuvette 3/4 full of crime scene wine.

8. Collect and rename the data for crime scene wine.
   a. Remove the cuvette of fresh wine and replace it with the crime scene wine. Make sure to align the cuvette correctly.
   b. Click or tap Collect. The absorbance *vs.* wavelength spectrum will be displayed. Click or tap Stop.
   c. To rename the run, click or tap More Options next to Data Set 2 on the table, then select Rename data Set.
   d. Enter **Crime Scene** for the name of this run. Click or tap RENAME.

9. Click on the y-axis label and select the data for Fresh Wine. Compare the two graphs. Do you notice any difference in the spectra? You will use your observations of differences when you answer the Case Analysis questions.

10. Prepare a cuvette of known poison by filling the cuvette 3/4 full with one of the known poison solutions.

11. Collect and rename the data for the poison.
    a. Remove the cuvette of crime scene wine and replace it with a known poison.
    b. Click or tap Collect.
    c. A full spectrum of the poison will be displayed. Click or tap Stop.
    d. To rename the run, click or tap More Options next to Data Set 3 on the table, then select Rename Data Set.
    a. Enter **Poison** for the name of this run, then click or tap RENAME.

12. Repeat Steps 10 and 11 procedures to collect the spectra for the rest of the known poisons. Make sure to rename all the spectra as you collect them.

13. Determine which poison is in the crime scene wine.
    a. Examine the spectra displayed on the screen. Change the graphs displayed by clicking or taping on the y-axis label and selecting the data sets you want to see.
    b. Look for a poison that has an absorbance peak at a wavelength similar to the anomaly you noted when you were comparing the Fresh Wine to the Crime Scene Wine in Step 9.

14. In the Evidence Record, note the poison you suspect was used in the crime scene wine.

15. Click or tap File (file menu icon), then choose Save. Name this file **Wine and Poison Spectra**.

Part II Determine the Concentration of the Poison

Use the poison solution that you determined from Part I for preparing the standards.

16. Prepare the crime scene wine sample and standard solutions.
    a. Label five clean, dry test tubes with numbers 1 through 5.
    b. The following table shows how much water and stock poison solution to add to each test tube. Use a pipette or a dropper and graduated cylinder to carefully measure the correct amount of poison solution into each test tube. **Note**: Use a separate pipette or graduated cylinder and dropper for the water and the poison.
    c. Carefully stir the contents of each test tube with a clean stirring rod. Clean the stirring rod with distilled water thoroughly and dry it with a tissue before using it in the next test tube.
    d. Label a sixth test tube **CS** for crime scene wine. Use a pipette or a dropper and graduated cylinder to measure 5 mL of crime scene poisoned wine into the test tube.

Test Tube	Poison Solution (mL)	Distilled Water (mL)	Final Concentration of Poison (mol/L)
1	10	0	0.015
2	8	2	0.012
3	6	4	0.0090
4	4	6	0.0060
5	2	8	0.0030

17. Prepare the cuvettes.
    a. For each standard solution, rinse an empty cuvette twice with about 1 mL of the sample.
    b. Fill each cuvette 3/4 full with the sample, and seal it with a lid.
    c. Label the lid with the test tube number.
    d. Wipe the outside of each cuvette with a tissue.
    e. Prepare a cuvette of crime scene wine. Label the lid **CS**.
    f. Prepare a cuvette of water for the blank. Label the lid **B**.

18. Click or tap File, 🗋, and choose New Experiment.

19. Select Absorbance *vs.* Concentration.

20. Calibrate the spectrometer as you did in Step 4.

21. Determine the optimal wavelength for creating the standard curve and set up the data-collection mode.
    a. Remove the blank cuvette, and place the 0.015 M standard, Cuvette 1, that you prepared in Step 16 into the cuvette slot.
    b. A spectrum of absorbance *vs.* wavelength will be displayed.
    c. Locate an absorbance peak in the spectrum and click or tap on that graph peak to select this wavelength
    d. Record the wavelength value being used on your Evidence Record.
    e. Click or tap Done.

22. You are now ready to collect absorbance-concentration data for the five standard solutions.

23. Click or tap Collect. Wait for the absorbance value to stabilize. Click or tap Keep. Type **0.015** in the edit box as the concentration in mol/L, then click or tap Keep Point. The data pair you just collected will now be plotted on the graph.

24. Place Cuvette 2 in the spectrometer, wait for the value to stabilize, and then click or tap Keep. Type **0.012** in the edit box as the concentration in mol/L, then click or tap Keep Point.

25. Repeat the procedure for Cuvette 3 (0.009 M), Cuvette 4 (0.006 M), and Cuvette 5 (0.003 M). **Note**: Wait until Step 28 to test the crime scene wine sample.

26. Stop data collection only after you have collected data for all the known samples.

27. Record the absorbance and concentration data values in the data table as part of your Evidence Record.

28. Place the cuvette with the crime scene wine in the cuvette slot in the spectrometer. Observe the absorbance value in the meter window. When the absorbance value has stabilized, round it to the nearest 0.001 and write it in your Evidence Record.

29. To determine the concentration of poison in the crime scene wine sample, fit a straight line to the graph of absorbance *vs*. concentration.
    a. Examine the graph of absorbance *vs*. concentration. To see if the curve represents a direct relationship between these two variables, click or tap Graph Tools, , then select Apply Curve Fit. A best-fit linear regression line will be shown for your five data points. Click or tap Apply.
    b. The linear-regression statistics for these two data columns are displayed for the equation in the form

$$y = mx + b$$

where x is concentration, y is absorbance, m is the slope, and b is the y-intercept. Record the values of m, b and correlation coefficient, r in your Evidence Record.

Note: One indicator of the quality of your data is the size of b. It is a very small value if the regression line passes through or near the origin. The correlation coefficient, r, indicates how closely the data points match up with (or fit) the regression line. A value of 1.00 indicates a nearly perfect fit. The graph should indicate a direct relationship between absorbance and concentration, a relationship known as Beer's law. The regression line should closely fit the five data points and pass through (or near) the origin of the graph.

30. Click or tap File, then choose Save. Name this file **Beers Graph**.
31. Discard the solutions as directed by your instructor. Proceed to Step 1 of Case Analysis.

EVIDENCE RECORD

Volume of the suspicious wine recovered from deceased victim's glass at crime scene: 73 mL

Identity of suspected poison: ________________________________

Wavelength used for this data: _____________

Solution number	Concentration of simulated poison in solution (mol/L)	Absorbance
1	0.015	
2	0.012	
3	0.0090	
4	0.0060	
5	0.0030	
CS	crime scene wine	

m	
b	
correlation, *r*	

Concentration of poison in the crime scene wine sample: ____________________________

CASE ANALYSIS

1. How did you determine that the crime scene wine had been poisoned?

2. Which poison was in the crime scene wine? How did you determine this?

3. Write the equation for the line in the form $y = mx + b$. Remember, y = absorbance, m = slope, x = concentration, and b = y-intercept. Use the values for m and b that you recorded in the Evidence Record.

4. Use the equation to calculate the concentration of poison in the crime scene sample.

5. Look up information on the poison you determined in Part I. The crime techs at the scene recovered a wine glass used by one of the victims with 73.0 mL of wine remaining in it. From interviews of the servers and by making measurements, the technicians determined that this wine glass has a total volume of 240 mL when filled. Assuming the victim started with a full glass and ingested the rest of the wine, and based on the calculated concentration of poison in the crime scene sample that you tested, determine the mass of the poison that was ingested. **Hint**: what information would you need to determine the mass of a compound from its concentration and volume?

6. Look up the LD_{50} (lethal dose that kills 50% of the population) for the poison you determined was used in the wine. You may need to get this value from your instructor. The victim who ingested the poisoned wine from the crime scene was a woman, 5 foot 5 inches tall, 110 lbs. Based on the mass of poison you calculated in Question 5, was this poison the cause of her death? Show all your work.

CASE REPORT

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

- Your initial findings about how you determined the wine was poisoned and how you determined the nature of the poison.
- How did you determine the concentration of the poison in the sample recovered from the crime scene?
- How did you determine if the poisoned wine ingested caused this victim to die?
- Graphs and supporting data.

Mystery Powder from a Crime Scene

A white powder is collected as evidence from 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, conductivity, and melting point of the powder. You will also observe the reaction with iodine solution, and an acidified ferric ion solution. After comparing the properties of the crime scene powder to those of known substances you will report what you have determined as 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

Chromebook, computer, or mobile device
Graphical Analysis 4 app
Go Direct pH
Go Direct Conductivity
Go Direct 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
mystery powder from the crime scene
iodine solution
0.2 M $Fe(NO_3)_3$ solution in HNO_3

PROCEDURE

Part I Chemical and Physical Properties

1. Obtain and wear goggles. **Caution**: *Be careful not to ingest any solid or solution, or spill any on your skin. Inform your teacher immediately in the event of an accident.*
2. Label 6 test tubes. Five will contain the known powders and one will contain the mystery powder from the crime scene.
3. Place enough distilled water in a test to tube to completely immerse the opening at the end of the Conductivity probe.
4. Fill all the rest of the test tubes with the same volume of distilled water.
5. Dissolve about 0.1 g of each known powder in each of the labeled test tubes. Stir each with a clean stirring rod. Note any reactions in your Evidence Record.
6. Dissolve about 0.1 g of mystery powder from the crime scene in the 6th, labeled test tube.
7. Launch Graphical Analysis. Connect the Conductivity Probe to your Chromebook, computer, or mobile device. A meter window can be accessed from the view icon.
8. Measure the conductivity of each solution and record the value in the Evidence Record. Rinse the Conductivity Probe with distilled water between measurements.
9. Disconnect the conductivity probe. Start a New Experiment from the File menu (file menu icon).
10. Connect the pH Sensor to your Chromebook, computer, or mobile device.
11. Measure the pH of each solution and record the values in the Evidence Record. Rinse the sensor in distilled water between measurements.
12. 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*.
13. Dispose of the waste as directed by your instructor. Rinse all the test tubes thoroughly.
14. Fill the test tubes with the same volume of water you used in step 4.
15. Place about 0.1 g of each powder in the test tubes as you did in steps 5 and 6.
16. 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*.
17. Record your observations in the Evidence Record.
18. Dispose of the waste as directed by your instructor. Rinse all the test tubes thoroughly.

Part II Determine the Melting Point

17. Obtain a small amount of the white powder assigned to you by your instructor. If your sample is not powdered, use a mortar and pestle to carefully grind the solid to a powder.

18. Check the control dial on the Melt Station to confirm that it is in the Off position. Connect the Melt Station power supply to a powered electrical outlet. Once connected to AC power, turn the control dial to the cooling setting.

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

20. Prepare a sample for melting.
    a. Pack a capillary tube 3–4 mm (~1/8 inch) deep with your 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 counter top to compress the sample into the closed end.
    d. Carefully insert the capillary tube of solid into one of the three slots in the heating block of the Melt Station. You may rotate the Melt Station toward you slightly for a better look at the heating block.

21. Start data collection.
    a. Click or tap on Collect.
    b. Turn the control knob on the Melt Station to Rapid Heat.
    c. Since you don't know the expected melting temperature, leave the dial on Rapid Heat and carefully observe the sample.
    d. Observe the graph of temperature *vs.* time.
    e. Note the temperature when the sample starts to melt and when it is completely melted. Record these values in the Evidence Record.
    f. When the sample has completely melted, click or tap Stop to stop data collection.
    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.
    h. Remove the capillary tube and dispose as directed by your instructor.

22. Conduct a second trial with a new sample of the same white powder in a new capillary tube.
    a. Click or tap Collect. **Note**: The previous data set is automatically saved.
    b. Turn the control knob on the Melt Station to Rapid Heat.
    c. Observe the graph of temperature *vs.* time.
    d. When the temperature is within 10–15°C of the expected melting temperature as determined in the previous step, turn the control knob to a point on the dial very near to the expected melting temperature.

23. Carefully observe your sample. In the Evidence Record, record two temperatures: (1) the temperature at which the sample particles first begin to melt and (2) the temperature when the complete sample has melted.
24. When the sample has completely melted, click or tap Stop to stop data collection.
25. Turn the control knob on the Melt Station to Off.
26. Calculate the range of melting temperatures for the second trial and record this in the Evidence Record.
27. Share the data for your sample with the rest of the class.

EVIDENCE RECORD

Part I Chemical and Physical Properties

Observations

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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 started melting temp (°C)	Rough final melting temp (°C)	Second started 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 might 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 might also be performed to improve your conclusion?

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