Case File 9

Killer Cup of Coffee:
Using colorimetry to determine concentration of a poison

Determine the concentration of cyanide in the solution.

A Killer Cup of Coffee? GlobalTech Manager Dies

SOUTH PAINTER, Tuesday: It was a normal Monday morning at GlobalTech Industries until the mail boy discovered project manager Patrick Marchand dead in his cubicle, head on his desk. Mr. Marchand had died while writing an email, in a room full of people hard at work. An early examination of the crime scene yielded no clues.

Mr. Marchand was known to have a serious heart condition, and many signs pointed to cardiac arrest as the cause of his death. However, as police canvassed the office space, the distinct odor of bitter almonds was detected, and a vial containing a small amount of an unknown chemical was found discarded in a communal trash can.

Based on the bitter almond odor, police have tentatively identified the substance as cyanide. The existence of this possible poison has lead police to suspect foul play in Mr. Marchand's death. The police have no leads.

continued on p. D2

This vial, wrapped in a piece of tissue, was discovered in the bottom of a communal trash can near the GlobalTech office bathroom. It once contained an unspecified amount of cyanide.

LAB NOTES

Technician: Beverly Chin

-received vial containing 20 mL cyanide solution—concentration unknown

-reacted cyanide (CN^-) solution with potassium polysulfide (K_2S_x) to produce potassium thiocyanate (KSCN)

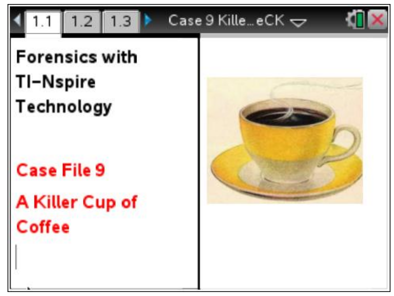
-reacted KSCN solution with iron(III)chloride ($FeCl_3$) to produce iron(III)thiocyanate ion ($FeSCN^{2+}$)

-determination of amount of $FeSCN^{2+}$ in reacted solution will allow estimation of concentration of CN^- in original solution

-included in package: $FeSCN^{2+}$ solution of unknown concentration

About the Lesson

- This lab introduces students to colorimetry. Students will calculate the concentration of an unknown by measuring how it absorbs a specific wavelength of light. The activity also demonstrates the importance of accurately made standard solutions.
- Teaching time: one 45 minute class period



Science Objectives

- Use Beer's law to determine the concentration of simulated iron(III) thiocyanate ($FeSCN^{2+}$) in an unknown solution.
- Use colorimetry to determine the concentration of a colored species in a solution.
- Use a linear relationship to model the data (Beer's law).
- Learn the importance of carefully prepared standards.

Activity Materials

- TI-Nspire™ technology
- *Case 9 Killer Cup of Coffee.tns* file
- *Case_9_Killer_Cup_of_Coffee_Student.doc* student activity sheet
- Vernier EasyLink™ or TI-Nspire Lab Cradle
- Vernier Colorimeter
- 7 cuvettes
- colored wax pencil
- distilled water
- waste beaker
- two 5 mL beakers
- stirring rod
- goggles
- 5 mL of simulated $FeSCN^{2+}$ solution of unknown concentration in the suspicious vial
- two 10 mL pipettes or graduated cylinders
- 50 mL of 0.15 M stock simulated $FeSCN^{2+}$ solution
- 2 droppers
- 6 test tubes
- test tube rack
- lint-free tissues

TI-Nspire™ Navigator™

- Send out *Case 9 Killer Cup of Coffee.tns* file.
- Monitor student progress using Class Capture.
- Use Live Presenter to spotlight student answers.

Teacher Notes and Teaching Tips

- The student activity sheet and .tns file contain the complete instructions for data collection. All assessment questions are also included in both places giving you the flexibility to either collect the .tns files with student data/answers (using TI-Nspire Navigator) or the student activity sheet.
- Use of the Colorimeter with the handheld is extremely battery intensive. It is recommended to keep handhelds charging between classes or using the lab cradle to minimize battery drain. Make sure that all solution preparation and cuvette filling is done *before* turning on the Colorimeter, in order to minimize the battery drain on the calculator.
- If class time is limited, prepare the solutions (Part 1) for the students before class.
- Before assigning the activity, you may want to review the visible spectrum of light and the concept of light absorbance. In addition, you may want to discuss the chemistry involved in the reactions. (See Background below.)
- The concentration and type of dye, as well as the size of the drops, can vary in different brands of food coloring. You may need to test several brands of food coloring in order to get consistent results. The stock solution directions and sample data below were obtained when Durkee® red food coloring was used.

Background

The primary objective of this experiment is to use a Colorimeter to determine the concentration of an unknown solution. In this device, blue light (470 nm) from the LED light source will pass through the solution and strike a photocell. We see the solution as a red color because the substances in the liquid are reflecting specific wavelengths of visible light and absorbing other wavelengths. Blue light is used in the test because the solution is absorbing those wavelengths, and the amount of blue light absorbed is proportional to the concentration of the substance in solution. A colored solution of higher concentration absorbs more light, and transmits less light, than a solution of lower concentration.

The reaction of the ferric ion (Fe^{3+}) and thiocyanate ion (SCN^{-}) produces the red brown solution simulated in this experiment. The table below shows the relationship between ion species and color.

$Fe^{3+}(aq)$	+	$SCN^{-}(aq)$	→	$FeSCN^{2+}(aq)$
yellow		colorless		red brown

Modifications

For less-advanced students, prepare the standard solutions (Part 1) in advance.

For more-advanced students, use several different unknowns. More-advanced students may also benefit from using qualitative chemical components. You can create stock solutions of KSCN and $FeCl_3$ or $Fe(NO_3)_3$ and have the students carry out the reaction in the Background Information section. Please note that even low concentrations of these chemicals give intensely colored solutions that may not give linear results when blue light is used. You may have to use very dilute solutions in order to get usable data.

Allow students to read the forensics scenario on the first page of their student activity sheet.

Procedure

Teacher Preparation of Solutions (Prior to the Lab)

1. You will be preparing a solution of red food coloring dissolved in water to simulate the cyanide-laced poison found at a crime scene. Food coloring is used so that preparation and disposal are easier, and safety issues with students handling the solutions are minimized.
2. Prepare the simulated 0.15 M $FeSCN^{2+}$ stock solution by adding 2 drops of red food coloring to 100 mL of distilled water.
3. To prepare an unknown simulated $FeSCN^{2+}$ solution that the test will determine is lethal, perform the following:
   Mix 7 mL of the simulated 0.15 M $FeSCN^{2+}$ stock solution with 3 mL of distilled water. This will give an unknown concentration of about 0.105 M. At this concentration, a volume of 44 mL of cyanide solution would have been needed to yield a fatal dose. An equivalent volume would be about three or four coffee creamers.
4. To prepare an unknown simulated $FeSCN^{2+}$ solution that the test will determine is not lethal, perform the following:

   Mix 1 mL of the simulated 0.15 M $FeSCN^{2+}$ stock solution with 9 mL of distilled water. This will give an unknown concentration of about 0.015 M. At this concentration, a volume of 308 mL of cyanide solution would have been needed to yield a fatal dose. This is more than an average size cup of coffee. It seems unlikely that anyone could have added that much solution to Mr. Marchand's coffee without him noticing.

Part 1 – Preparing the Solutions
Move to pages 1.2–1.7.

Students will need to follow directions on pages 1.2-1.7 to prepare the solutions for testing.

Optional: If you have limited class time, you can prepare the solutions in advance of the lab. Be sure to tell students to skip Part 1 in their activity sheet and .tns file, if you prepare these before.

Directions for the Advance Preparation of the Solutions:

1. Obtain and label the following with a wax pencil:
   a. Pour 50 mL of stock simulated 0.15 M $FeSCN^{2+}$ solution into a 50 mL beaker. Label the beaker "Simulated 0.15 M $FeSCN^{2+}$."
   b. Pour 30 mL of distilled water into a 50 mL beaker. Label the beaker "H_2O."
2. To prepare the unknown and standard solutions in advance:
   a. Label five clean, dry test tubes with numbers 1 through 5.
   b. The following table shows how much water and stock simulated $FeSCN^{2+}$ solution to add to each test tube. Use a pipette or a dropper and graduated cylinder to measure the correct amount of simulated $FeSCN^{2+}$ solution into each test tube. **Note:** Use a separate pipette or graduated cylinder and dropper for the water and the simulated $FeSCN^{2+}$.
   c. Carefully stir the contents of each test tube with a clean stirring rod. (Clean the stirring rod with distilled water and dry it thoroughly in between each test tube.)
   d. Label a sixth test tube with a "U" for unknown. Use a pipette or a dropper and graduated cylinder to measure 5 mL of simulated $FeSCN^{2+}$ solution of unknown concentration into the test tube.

Test Tube	$FeSCN^{2+}$ Solution (mL)	Distilled Water (mL)	Final Concentration of $FeSCN^{2+}$ (mol/L)
1	10	0	0.15
2	8	2	0.12
3	6	4	0.09
4	4	6	0.06
5	2	8	0.03

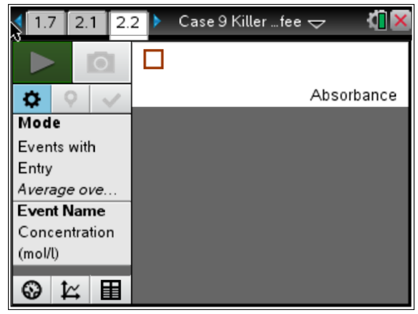
3. To prepare the blank, the five standard solutions, and the unknown for colorimetry:
   a. For each standard solution, rinse an empty cuvette twice with about 1 mL of the sample.
   b. Fill each cuvette 3/4 full with the sample, and seal it with a lid.
   c. Label the lid with the sample number.
   d. Wipe the outside of each cuvette with a tissue.
   e. Repeat Steps 3a–3d for the unknown sample. Label the lid with a "U".
   f. Repeat Steps 3a–3d using distilled water for the blank. Label the lid with a "B".

Remember the following:

- All cuvettes should be clean and dry on the outside.
- Remind students to handle cuvettes only by the top edge or the ribbed sides.
- All solutions should be free of bubbles.

Part 2 – Collecting Data

Move to pages 2.1-2.4.

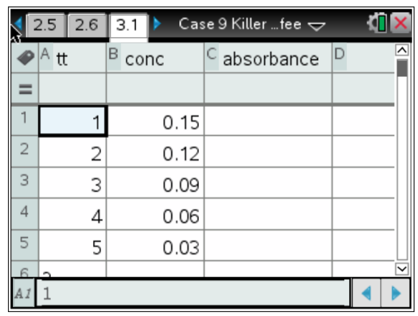


4. Students will enter the volume of the suspicious vial in the Evidence Record. Assist students in connecting the colorimeter to TI-Nspire, calibrating the colorimeter, and then collecting absorbance-concentration data.

   In the Evidence Record, students write down the absorbance values displayed in the Data Table on page 2.2. At this point, students will have not done the unknown!

Part 3 – Analyzing the Data

Move to pages 2.5-3.3.



To determine the concentration of $FeSCN^{2+}$ in the unknown solution, students use linear regression to fit a straight line to the graph of absorbance vs. concentration.

EVIDENCE RECORD

SAMPLE DATA

Volume of the suspicious vial **20 ml**

Solution Number	Concentration of $FeSCN^{2+}$ in Solution (mol/L)	Absorbance
1	**0.15**	**0.623**
2	**0.12**	**0.486**
3	**0.09**	**0.365**
4	**0.06**	**0.243**
5	**0.03**	**0.128**
?	Unknown	**0.436**

Concentration of $FeSCN^{2+}$ in the unknown solution **0.105 mol/L**

y	*mx* + *b*
m	**4.11**
b	**-9×10^{-4}**
correlation	**0.9995**

Case Analysis

Have students answer the following questions in the .tns file, on their activity sheet, or both.

Q1. Write the equation for the line in the form $y = mx + b$, using the values for m and b that you recorded in the Evidence Record. For example, if $m = 3$ and $b = 6$, then the equation for the line is $y = 3x + 6$.

Answer: Answers will vary. For the sample data, $y = 4.11x - 9 \times 10^{-4}$.

Q2. Use the equation to calculate the concentration of $FeSCN^{2+}$ in the unknown solution. How does the value you calculate compare with the value you read from the graph?

Answer: Answers will vary. For the sample data, $y = 4.11x - 9 \times 10^{-4}$.

$$x = \frac{y + 9 \times 10^{-4}}{4.11} = \frac{0.436 + 9 \times 10^{-4}}{4.11} = 0.106 \text{ mol/L}$$

The equation and the graph yield almost the same value for the FeSCN2+ concentration.

Q3. The volume of the cyanide solution that was found at the scene was 20 mL. Based on the calculated concentration of $FeSCN^{2+}$ in the unknown solution, determine the concentration of potassium cyanide, KCN, in the original poison. Show all of your work. Give your answer in milligrams of KCN per milliliter of solution. **Hint:** One mole of KCN will produce one mole of $FeSCN^{2+}$. Assume that all of the KCN in the poisoned solution reacted to form $FeSCN^{2+}$. Assume that the 20 mL of original solution was not diluted during the reaction to form $FeSCN^{2+}$ and that the sample you received was also undiluted. The molecular weight of $FeSCN^{2+}$ is 114 g/mol. The molecular weight of KCN is 65 g/mol.

Answer: Answers will vary, depending on the concentration of $FeSCN^{2+}$ that you use in the unknown. A sample calculation for an unknown concentration of 0.106 mol/L is given below:

$$\frac{0.106\ \text{mol FeSCN}^{2+}}{1\ \text{L solution}} \times 0.020\ \text{L solution} = 0.00212\ \text{mol FeSCN}^{2+}$$

$$= 0.00212\ \text{mol KCN in original solution}$$

$$0.00212\ \text{mol KCN} \times 65\ \text{g/mol} = 0.138\ \text{g KCN in original solution}$$

$$= 138\ \text{mg KCN}$$

$$\frac{138\ \text{mg KCN}}{20\ \text{mL solution}} = 6.9\ \text{mg KCN/mL solution}$$

Q4. For most people, swallowing 300 mg of KCN is fatal. Based on the concentration of KCN in the poison that you calculated in Question 3, determine the approximate volume of poison that the victim would have to have swallowed for it to have killed him. Show all of your work.

Answer: Answers will vary depending on the concentration of KCN in the original solution. A sample calculation for a 6.9 mg/mL KCN solution is given below:

$$\frac{1\ \text{mL solution}}{6.9\ \text{mg KCN}} \times 300\ \text{mg KCN} = 44\ \text{mL solution}$$

Q5. Is it likely that the poison was the direct cause of death? Explain your answer. **Hint:** Remember that the vial was mostly empty and may, at one time, have held more than 20 mL.

Answer: In this example the 20 mL bottle would not contain enough poison to cause death.

Q6. Suppose you found out that the concentration of $FeSCN^{2+}$ in the unknown was actually very different from the value you calculated in Question 2 and the value you read off the graph. What factors could have caused that to happen?

Answer: Factors that can cause error include inaccurately prepared standards, an uncalibrated or improperly calibrated Colorimeter, equipment error, impurities in the standard or unknown solutions, and mistakes in following the procedures.