Name:_____Class:_____Date:_____

The Case of Kirsten K.:
The Drug Lab Evidence

All Reports are to be filed using the correct format, following the Lab Report Style Guide

Part I: IR-Spectra Analysis

Case Background

After further investigation, police have uncovered what they think to be a drug lab in the basement of the lake house where the murder of Larry J. took place. Reports indicate that Gerald V. was frequently seen entering the cellar door that leads to the drug lab. Police uncovered the following:

- 3 "substances" in baby food jars labeled aspirin, caffeine, and acetaminophen
- A large Rubbermaid tub filled with 2240g coffee grounds
- Multiple bottles of what appears to be some sort of unknown drug

The police suspect the aspirin, caffeine, and acetaminophen were being used to create this unknown drug, and that someone was extracting the caffeine from coffee. Additionally, the police found a small spiral-bound notebook with a list of names, dates, and transactions. The police have determined that this list consists of people who bought the drugs made in this lab. One of the names on the list was Larry J.. Finally, Gerald's fingerprints were found on the baby food jars and on the bottles of unknown drugs.

The CSI unit of the local Police Department is requesting your help in order to solve this crime once and for all. It is up to you to assemble these final pieces of evidence, determine who should be charged with the murders of Kirsten K. and her husband, Larry J., and also present if there are any additional charges the killer(s) could be facing. Good Luck! (Be sure to tie up any loose ends still remaining from other parts of the investigation).

In order to acquire detailed chemical information about the substances found in the baby jars, police investigators took the samples to the forensics lab, where they ran some IR spectra analysis. Unfortunately, the officer carrying the file of the spectra dropped the folder, and the IR spectra got mixed up. You need to determine which IR spectrum belongs to each of the three substances in the baby food jars.

Materials

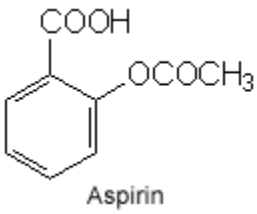
- IR Spectra of three substances (acetaminophen, aspirin, caffeine)

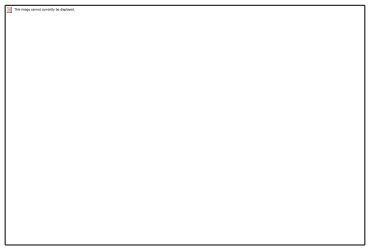
Data

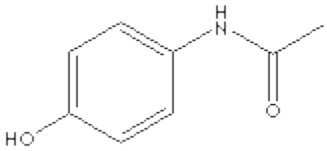
1. Research has been completed about the three substances (aspirin, caffeine, and acetaminophen). What are their empirical formulas?

	Molecular formula	**Empirical formula**
Aspirin:	$C_9H_8O_4$	_____
Caffeine:	$C_8H_{10}N_4O_2$	_____
Acetaminophen:	$C_8H_9NO_2$	_____

2. Clearly circle and identify all organic functional groups present in each of the molecules.







Aspirin Caffeine Acetaminophen

3. Use the research you have collected to determine which IR spectrum from the evidence envelopes belongs to each of the substances. Create a data table below that shows which peaks for each spectra correspond to which functional groups and wavelengths.

Part II: Caffeine Extraction

Background Information

Police want to know if caffeine can actually be extracted from the coffee grounds, and if so, how much total caffeine could be extracted from all of the coffee grounds found in the Rubbermaid container in the basement of the lake house.

Purpose

To determine if caffeine can be extracted from coffee grounds

Procedure

1. To a clean 500mL Erlenmeyer flask, add 100mL of coffee extract. This extract is the solution that was prepared in advance by the instructor.
2. Add approximately 2 grams of sodium carbonate to the coffee solution. This will react with some of the substances in the coffee extract and make them extremely water soluble. Swirl the mixture until all the sodium carbonate dissolves.
3. Add 25mL of ethyl acetate and vigorously swirl the mixture for 10 minutes. Do not shake the mixture.
4. Allow the mixture to stand and separate into 2 layers, a dark aqueous bottom layer and a clear ethyl acetate top layer.
5. Carefully pour off as much of the clear layer as possible into a beaker, being careful not to remove the bottom layer. This process is called decanting.
6. Place a 12.5cm fluted filter paper in a long stem glass funnel. Put the funnel in a small iron ring and suspend it over a 250mL Erlenmeyer flask.
7. Using a squeeze bottle of water, thoroughly wet the filter paper.
8. Slowly and carefully pour the ethyl acetate/water mixture into the fluted filter paper. The excess water will drain through and the ethyl acetate solution of caffeine will remain on the filter paper.
9. Using a pipet, transfer the ethyl acetate solution to a 50mL Erlenmeyer flask. To this solution, add a scoop of anhydrous sodium sulfate in order to remove the last traces of water.
10. While the solution is drying, weigh a 50mL beaker to the nearest 0.01g. Record this in the data table. Tare the scale.
11. Using a pipet, transfer the dried solution to the 50mL beaker.
12. Evaporate most of the ethyl acetate in the hood on a warm hot plate. When only a faction of a milliliter of liquid is left, remove the beaker from the hot plate. Allow the beaker to stand in the hood for a minute or two. The heat remaining in the glass will cause the last amount of ethyl acetate to evaporate and produce a solid residue of crude caffeine.
13. In order to determine your recovery of caffeine, reweigh the cool beaker and record this mass in the data table.

Materials

- Coffee solution
- 500mL Erlenmeyer flask
- 250mL beaker
- Filter paper
- Iron ring
- Solid sodium bicarbonate
- Balance
- Pipet
- Hotplate
- Methylene chloride/ethyl acetate
- Funnel
- Anhydrous sodium sulfate
- 50mL beaker

Data

***Instructor Notes: Coffee extract solution prepared with 50 g of coffee/1000mL

Caffeine Extraction Data

Mass of Empty 50mL beaker	
Mass of 50mL beaker and Caffeine	
Mass of Caffeine	

Conclusion/Discussion Questions

1. Were you able to obtain caffeine crystals from the coffee ground solution?

2. How much total caffeine could be extracted from all of the coffee grounds found in the basement?

3. Describe the appearance of your product. Caffeine is a white solid, but your product was probably discolored. Why?

Part III: Thin Layer Chromatography of an Unknown Drug

Background Information

Thin Layer Chromatography, or TLC, is one technique that is commonly used to identify unknown drugs. You will use TLC to identify the components contained in the bottles of unknown drug found at the scene. You will use the three substances (aspirin, caffeine, and acetaminophen) as your known solutions to determine if any of them are also apparent in the unknown drug.

Purpose

To determine the components in the unknown drug

Procedure

1. Cut appropriate size chromatography paper (if not already cut for you). Handle the paper by the edges.
2. Mark a pencil line across the bottom of the chromatography paper and label a spot for each solution tested.
3. Apply 6-8 dots of each solution onto the TLC paper (3 known and 1 unknown) using capillary tubes to the labeled spots on the pencil line.
4. Add solvent (ethyl acetate) to the beaker/bottle. Make sure the line with the solution spots will not be submerged in the solvent.
5. Run the solvent at least 6-7cm up the paper.
6. Mark a solvent front (or the height to which the solvent went) with a pencil.
7. Measure the distance the solvent moved and the distance each component of the solutions moved.
8. Calculate the R_f values for each of the solutions in the Analysis section.

Materials

- Development jar with lid
- TLC plate, 4” x 2”
- Aspirin standard solution, 6-10 drops
- Acetaminophen standard solution, 6-10 drops
- Caffeine standard solution, 6-10 drops
- “Unknown” solution
- UV light source
- Ethyl acetate
- Microcapillary tubes, 4
- Ruler
- Pencil

Data

Design a data table that shows the distance traveled for the compound and the solvent, as well as R_f values for each of the known substances and the unknown drug.

Analysis—Calculations

Use the space below to show R_f value calculations based on the equation given below.

$$\text{Rf value} = \frac{\text{distance traveled by compound}}{\text{distance traveled by solvent}}$$

Conclusion

1. What component(s) were present in the unknown drug?

Part IV: Thin Layer Chromatography of Pen Matching

Background Information

The police have acquired pens from each of the suspects and the new victim, Larry J.. They have run an ink analysis on the information found in the spiral notebook. You will perform a chromatography test on each of the pens to see if any of them match the ink from the drug log book where Larry J.'s name was found recorded several times.

Purpose

To determine whose pen can be matched to the ink from the drug log.

Procedure

1. Cut the filter paper into 3 strips. Label with a pencil at the top of each piece of filter paper, which suspect the pen belongs to.
2. Make a horizontal mark (about 1 in from the bottom) with one of the pens across one of the pieces of filter paper. Repeat this with each pen and each piece of filter paper.
3. Pour the solvent into the bottom of each of the beakers so that the solvent does not reach the mark made by the pen.
4. Get the evidence folder and try to match the evidence filter papers with those from the pens from each of the suspects.

Materials

- Suspect pens (Gladys, Gerald, Larry)
- Ink from drug log
- Coffee filters or filter paper
- Fingernail polish remover (acetone)

Data

Construct a data table to record any qualitative and/or quantitative observations about each of the suspect samples compared to the evidence samples.

Conclusion

1. Which pen(s) match the ink samples from the drug log book?

2. What can you conclude, if anything, from the new information presented in relation to the crime?

THE FINAL SCENARIO

Record the final scenario, linking all of the assessments together and evidence to support all theories in the case. Who should be convicted and for what crimes?

Kirsten K. Murder Case

Larry J. Murder Case