Fall 2015

Chemistry 227H

Pre-lab assignments and experimental procedures

Instructor: Dr. Johansson

TA: Colin Hiatt
Chemistry 227H  
Schedule – Fall 2015

Week 1: 9/29 and 10/1

1. Check-in.  2. Lab Safety and 3. Avogadro Case Study  

Week 2: 10/6 and 10/8

Chromatography / Filthy Lucre

Week 3: 10/13 and 10/15

Analyze GC/MS data

Week 4: 10/20 and 10/22

Copper Cycle

Week 5: 10/27 and 10/29

Painting Case Study – this is a computer lab

Week 6: 11/3 and 11/5

Pigment analysis

Week 7: no Tuesday lab  11/12 only

Atomic Emission

Week 8: 11/17 and 11/19

Molecules and Light – Natural Dyes – Beer’s Law dye absorption

Week 9: 11/24 only (no Thursday lab Thanksgiving Holiday)

Atomic Emission Spectra

Week 10

Check out. (Make Up Lab - only for excused absences)
1 Laboratory safety rules and procedures

This is the very first section in the laboratory manual because it is the most important section in the laboratory manual. SAFETY FIRST!

1.1 Safety rules

The guidelines below are established for you and your classmates’ personal safety. Failure to adhere to the guidelines below will result in a loss of Lab Technique points.

- Personal Protective Equipment (PPE) is used to protect you from serious injury and/or illness resulting from direct contact with chemical hazards in the laboratory. Spills and other accidents can occur when least expected, and you may not be aware that someone else has spilled something. For this reason it is necessary to wear proper PPE at all times. The PPE for student labs consist of goggles, gloves and clothing. Proper PPE is required for all students or they will be asked to leave the lab.

- Goggles protect your eyes. They must be worn at all times in the laboratory. Only indirect-vented goggles are allowed in the student labs. These are for sale in the bookstore and stockroom. You should not wear contact lenses in a chemical laboratory. Chemical vapors may become trapped behind the lenses and cause eye damage. Some chemicals may dissolve contact lenses. The most important aspect of having the goggles fit comfortably is the proper adjustment of the strap length. Adjust the strap length so that the goggles fit comfortably, securely and are not too tight. If you find that your goggles tend to fog, you can pick-up anti-fog tissue from the stockroom.

- Gloves should be worn to protect the hands from chemicals. Gloves are provided through your student fees and are located in the student labs. For health and safety reasons it is important to always remove at least one glove when leaving the student laboratory, this prevents things such as door handles from getting contaminated. It is your responsibility to ensure that those outside the laboratory are not exposed to contaminants carried by your gloves.

- Dress appropriately for laboratory work. You must wear shoes that cover your entire foot, including the heel. They should fit up near your ankle; leather is preferred but any non-porous material is okay. One of the most common laboratory accidents is glass piercing a body part. You do not want a broken pipette inside your foot just because you chose to wear sandals. Your clothing must cover your torso and legs down to your knees. Short shorts, short skirts, tank tops and halter tops are not allowed.

- Eating, drinking and smoking are prohibited in the laboratory at ALL times (of course). Wash your hands after finishing lab work and refrain from quick trips to the hall to drink or eat during lab. If you take a break, be certain to remove gloves and wash hands before ingesting food or drink, or before visiting the restroom.

- Never work alone in the laboratory or in the absence of the instructor.

- Headphones may not be worn in lab.
1.2 Safety procedures

- Know the location of safety equipment including fire extinguisher, fire blanket, first aid kit, safety shower, eyewash fountain and all exits.
- In case of fire or accident, call on the instructor immediately.
- Small fires may be extinguished by wet towels.
- If a person’s clothing catches fire, roll the person in the fire blanket to extinguish the flames.
- In case of a chemical spill on the body or clothing, stand under the safety shower and flood the affected area with water. Remove clothing to minimize contamination with the chemical.
- If evacuation of the lab is necessary, leave through any door that is safe, or not obstructed; doors that lead to other labs may be the best choice. Leave the building by the nearest exit and meet your TA on the field next to Hoffmann Hall. This would also be the meeting place in the event of an earthquake or other emergency. It is good to know the nearest exits of your lab on the first day of class.
- Spilled chemicals must be cleaned up immediately. If the material is corrosive or flammable, ask the instructor for assistance. If acids or bases are spilled on the floor or bench, neutralize with sodium bicarbonate, then dilute with water. Most other chemicals can be sponged off with water.
- Avoid contact with blood or bodily fluids. Notify the instructor or stockroom personnel if ANY blood is spilled in the lab so that proper clean up and disposal procedures may be followed.
- If a mercury thermometer is broken, do not attempt to clean up yourself. Notify students around you, so that mercury is not spread, then notify your lab instructor or stockroom personnel. The stockroom is equipped for proper clean up and disposal of mercury.

1.3 General Laboratory procedures and protocol

- Leave all equipment and work areas as you would wish to find them.
- Keep your lab-bench area neat and free of spilled chemicals. Your book bag, coat, etc., should be kept in the designated area at the entrance to the lab, not at your bench.
- All chemical waste must be disposed of in proper containers. Proper disposal of chemicals is important for student safety and proper disposal. Putting chemicals into the wrong containers can lead to injury from unexpected chemical reactions. Mixing waste can also make it more difficult or expensive for PSU to dispose of them. Only chemicals should go into waste jars. Waste jars for each experiment will be provided in the lab. They will be labeled specifying which contents should be placed inside. It is important that you replace the lids to the waste containers. When done with the waste jar, make sure it is placed in a secondary container. Do not put anything down the sink unless you are explicitly told to dispose of it this way. Your instructor will provide specific disposal guidelines when
needed. Following these guidelines assists us in lowering the environmental impact of the labs.

• There are several locations for very specific waste:

  **Chemical waste** These containers are ONLY for chemical waste generated in the lab. They are each specifically labeled for each lab and waste type. READ THE LABELS.

  **Contaminated paper waste** This is ONLY for paper towels used for clean-up of chemical spills.

  **Broken glass** This is ONLY for broken glassware.

  **Gloves** This is ONLY for used gloves.

  **Normal trash** This is for all other trash that is not chemically contaminated, glass, or gloves.

• Clean your bench, equipment, and glassware; dirty glassware is harder to clean later. Wash with water and detergent scrubbing with a brush as necessary. Rinse well with water. Do not dry glassware with compressed air, as it is frequently oily. The water and gas should be turned off and your equipment drawer locked.

• Clean the common areas before you leave the lab. Point deductions for the entire class will be imposed if the instructor or stockroom is not satisfied.

• Return any special equipment to its proper location or the stockroom.

**Handling chemicals**

• Read the label CAREFULLY. The Chemicals are organized by experiment in secondary containment bins. Make sure the chemical name and concentration match what is required by the experiment!

• Do not take the reagents to your bench.

• We recommend always picking up bottles by the label. If all students do this, then any un-noticed spills when pouring will not cause possible problems for the next user. Remember to wear gloves while working with reagents.

• Do not put stoppers or lids from reagents down on the lab bench. They may become contaminated. Be sure that the lids or stoppers are replaced.

• Do not place your own pipet, dropper, or spatulas into the reagent jar. Pour a small amount into a beaker and measure from that. Please pour on the conservative side to minimize waste and cost of labs. You can always go back for more.

• Do not put any excess reagent back in the reagent jar. Treat it as waste and dispose of it properly.

• Never weigh directly onto the weighing pan when weighing chemicals on the balances. Weigh into a weighing boat or beaker. Any spills on the balances MUST be cleaned up immediately. If you are unclear how to clean a spill, notify your instructor. The balances you are using are precision pieces of equipment and costs up to $4,000.
• All chemicals should be treated as potentially hazardous and toxic. Never taste a chemical or solution. When smelling a chemical, gently fan the vapors toward your nose.
• Any chemicals that come in contact with your skin should be immediately washed with soap and copious amounts of water.

Safe Practices
• Never pipet any liquid directly by mouth! Use a rubber bulb to draw liquid into the pipet.
• Never weigh hot chemicals or equipment.
• When heating a test tube, always use a test tube holder and be certain never to point the open end of the test tube toward yourself or another person.
• Handling glass tubing or thermometers: to insert glass tubing into a rubber stopper, lubricate the glass tubing with a drop of glycerin, hold the tubing in your hand close to the hole, and keep all glass pieces wrapped in a towel while applying gentle pressure with a twisting motion.
• To prepare a dilute acid solution from concentrated acid, acid should be added slowly to water with continuous stirring. This process is strongly exothermic, and adding water to acid may result in a dangerous, explosive spattering.
• Use the fume hood for all procedures that involve poisonous or objectionable gases or vapors.
• Never use an open flame and flammable liquids at the same time.
2 Pre-lab, in-lab, and post-lab

2.1 Keeping a lab notebook

In keeping a lab notebook, there are certain principles that should be followed. These boil down to being clear and complete in your entries in your lab notebook. There are also certain conventions for lab notebooks that are universally followed. High on this list are the following:

- Use a notebook with pre-numbered pages.
- Record entries in ink.
- Keep entries reasonably neat and organized.
- Never tear pages out of your lab notebook (other than the carbonless copy pages).

What Kind of Notebook Should I Use? For this class you must use a notebook with carbonless copy pages.

General Guidelines

- Write your name on the outside front of the notebook.
- Use black ink, fine-tipped ball-point pen (this will photocopy clearly).
- At the front of the notebook, leave a few pages for a Table of Contents.
- Generally use only the right hand page for most text.
- Use facing left page for working graphs, manual calculation, and working notes.
- Prepare data tables in advance - with columns for calculated results and notes.
- Working graphs are done in lab notebook to monitor progress.

Usage and structure The overriding principle for a lab notebook is to record in it all the pertinent information about your lab work. This boils down to clear descriptions of what you did and what you observed as a result. It is a working tool, and a reference for other researchers who might want to read your notebook and reproduce your work. (This applies to notebooks in learning laboratories: Your lab instructor may want to look at what you did in order to understand your results. This is often the case. So, it needs to be clear.) The word clear here is crucial. In order to be clear, data must be recorded in well-thought-out tables, clearly labeled. Descriptions of procedures must be clear and concise; to the point. You should record all your work in your lab notebook. That is the proper place for all lab planning and observations. Nothing should be recorded on odd scraps of paper, etc.

2.2 Pre-lab

Every pre-lab should contain two things:
1. Answers to all pre-lab questions (if pre-lab questions exist).

2. An experimental procedure that is detailed enough that your laboratory partner can use it to complete the laboratory assignment without consulting the laboratory manual. *You may not use the lab manual in the lab.* Specifically, the experimental procedure should have the following sections:

**Title** With your lab notebook laid open, on the right hand page write down the title of the experiment, and the date. In general, you will use the right-hand page for all your writing. The left-hand page is reserved for recording scratch work. Don’t use this space until you need to. One example of how to use the left-hand page: if your work requires simple calculations using your measurements, use the left-hand page to do the calculations. If unexpected results occur later, sometimes you can look back at your scratch work and discover the error. (Oh, I subtracted wrong! We put in 10.5 grams of copper sulfate, not 9.5 like we thought!) Better to discover the error after the fact than never to discover it at all.

**Procedures and data tables** This next section will be labeled Procedures and data tables. As the name suggests, write down what you will actually do and leave room for what you will observe. This section is where you should have pre-prepared tables for data collection. Set up this section by dividing the page into a right and left column. In the left hand column write your procedure and in the right column next to the procedure, leave space to record observations and data or measurements. Make data tables in line at the end of the procedures section.
2.2.1 Example of a prepared notebook

How Much Sugar is in a Can of Coke?

Purpose: The purpose of this experiment is to determine the mass (in grams) of sugar in a can of Coke using a calibration curve prepared using sugar-water solutions. The density of these solutions is related to the sugar content.

Procedure

1. Weigh 100 ml volumetric flask + stopper
2. Weigh ~2 g of sugar into a beaker
3. Transfer sugar to flask
4. Weigh flask + stopper + sugar
5. Add water until 1/3 way to the fill line
6. Dissolve sugar (no shaking)
7. Add water to fill line & mix
8. Repeat steps 1-7 to make solutions with ~4, 8, 12 g of sugar

<table>
<thead>
<tr>
<th>MASS IN GRAMS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flask</td>
</tr>
<tr>
<td>Flask 1</td>
</tr>
<tr>
<td>Flask 2</td>
</tr>
<tr>
<td>Flask 3</td>
</tr>
<tr>
<td>Flask 4</td>
</tr>
<tr>
<td>Flask 5</td>
</tr>
</tbody>
</table>

NOTE: INSERT PERIODIC TABLE UNDER COPY SHEET BEFORE WRITING • THE HAYDEN-McNEIL STUDENT LAB NOTEBOOK
2.3 In-lab

Use the pre-lab to do the experiment. Record all experimental details and observations (use the empty tables made in your procedure to record data you collect). Please note that you will not be allowed to consult the laboratory manual during lab hours. Part of you pre-lab preparation

<table>
<thead>
<tr>
<th>Procedure</th>
<th>Data/Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>9. Weigh 100 ml volumetric flask + stopper</td>
<td></td>
</tr>
<tr>
<td>10. Fill the flask to the fill line with flat coke</td>
<td></td>
</tr>
<tr>
<td>11. Weigh the flask + coke + stopper</td>
<td></td>
</tr>
</tbody>
</table>

Results/Calculations:
for all labs is always to write your own experimental procedure. This is not busy work. If you have already planned the experiment when you enter the laboratory you’ll find it much easier to complete the experiment.

2.4 Post-lab

For most experiments performed this term you will turn in a report that, unlike CH227H, must be typed. The reports are due at the beginning of class the week following completion of the experiment. Below is a description of what should be included in each section. The sections are presented here in the order they should appear in your lab report. It is expected that you will complete each experiment and do the necessary calculations and analysis during the scheduled lab period each week. You may discuss the calculations and analysis with your lab mates. Your written lab report should be your own individual work!! The lab reports for this course will be short and to the point. However, they are expected to be of highest quality, of course. Every post-lab should contain two things:

1. Answers to all post-lab questions (if post-lab questions are asked).
2. A writeup that presents your data and conclusions.

Use this guide to write your post lab (not all post-labs will need to contain all items):

Title  This is the title of the lab as given in the laboratory manual.

Purpose  The purpose is a condensed summary of the report’s findings with an emphasis on educational purpose of the laboratory exercise and should be written in the third person, passive voice. Write the purpose last. The purpose should be clear, concise, and self-contained, and only a few sentences long. Answer the three questions below to make the first version of the purpose. Finalize the purpose by word-smithing to make the sentences into a coherent paragraph.

1. What was done?
2. How was it done?
3. What was the outcome?
4. What aspect of chemistry was the lab designed to teach?

Data and data tables  Report collected data. Since you’re describing something you did in the past, this section should be written using past tense. Whenever possible, data should be presented in the clearest format possible, usually in the form of a table. When you present your data in a table it is necessary to take the following into account.

• Number tables sequentially as they appear (Table 1, Table 2, etc.).
• Be sure to refer the reader to view the tables in the text.
• Construct a descriptive table caption and place it above the table.
• Tables should include descriptive column headings, including units.
• Tables should not be divided across page boundaries.

For graphs, similar considerations apply:

• Number figures sequentially as they appear (Figure 1, Figure 2, etc.).
• In your writing, be sure to cite the tables in the text.
• Insert a caption below the graph that indicates what is being plotted on the y-axis vs. what is being plotted on the x-axis (always y vs. x)
• Each axis should be clearly labeled, including units.
• Figures should not be divided across page boundaries
• Remove gridlines, titles and equations from the graph. If this information is pertinent, it should be included in the caption.
• If the slope or intercept is necessary for other parts of the experiment, then place the values in the caption with proper units.

Observations and calculations  This is where you present observations and calculated data. If you did any calculations they are to be explained here.

1. Are calculations explained?
2. Units? Significant Figures?

Conclusions  This is where you draw conclusions that are supported by your experimental data. Writing the Conclusions section apart from the Data and data tables and Observations and calculations may make it seem clunky and sometimes a little repetitive but it is a way of communicating what you (the scientist) learned from doing the experiment. The Data and data tables contains only things you can swear to on the witness stand is correct. In theory, you can be wrong in the Conclusions but of course you don’t want to be.

1. Did you accomplish the purpose?
2. Did you only make logical deductions based on the results?
3. Have you discussed sources of error or ambiguities in the data?
4. Do your conclusions clearly contribute to the understanding of the overall problem?

General tips

1. Have you listed your name, your partner’s name, a descriptive lab title and date?
2. Did you check your spelling?
3. Is your report written in passive third person voice (you did not use the words I, we, they, etc.)
4. Is the proper tense is maintained within sections?
5. Have you correctly written chemical names, formulas, and balanced equations?
6. Were correct subscripts, superscripts, and symbols used?

7. Did you separate the numbers from their units (0.25 mL was added, not 0.25mL was added)?

8. Did you check significant figures?

9. Do your numbers include leading zeros (0.25 mL was added, not .25 mL was added)?

10. Did you make sure that you did not start a sentence with a number?

11. Are your references cited in an official style?

12. Have you made a citation whenever using ideas from the outside?

13. Are all subjects and verbs in agreement?

14. Did you make sure that there are no run-on sentences or fragments?

2.5 Grading criteria

Unless otherwise noted, every lab report is worth 55 points. Each report will be graded according to the following point distribution:

- Title (5 points)
- Purpose (10 points)
- Data and data tables (10 points)
- Observations and calculations (15 points)
- Conclusions (15 points)

In addition, an additional 45 points will be assigned each lab meeting based on your laboratory notebook and lab technique. At the end of each lab you must check out with your TA so that person can assess your lab notebook and verify that you have cleaned your work area.

**Notebook:** The TA can award up to 30 points based on the quality of your lab notebook. Your TA will be looking to see that you are including the items outlined in the pre-lab.

**Lab technique:** The TA can award up to 15 points based on the quality of your lab technique and methods, safety, general mannerism in lab and cleanliness.
Lab Safety Quiz

Name____________________________________

After watching the ACS safety video (a link is provided on D2L), print this document, complete the quiz and turn this in to your TA at the beginning of your second lab session.

1. I have watched the ACS safety video.
   o True
   o False

2. Do what you ought to; add the "concentrated" acid to the water....and not the other way around.
   o True
   o False

3. When pouring from a stock solution bottle, where should you hold onto the bottle?
   o The label
   o Make someone else pour it for you
   o The neck
   o The bottom

4. What does a "volatile" chemical refer to?
   o One that is a known carcinogen.
   o One that is explosive and should not be used.
   o One that produces vapor and should be stored in the fume hood

5. To keep the general chemistry laboratory "green", it is important to:
   o Recycle any unused stock solutions by carefully pouring any unused solution back into the stock bottle.
   o Take only slightly more of the stock solution than needed and properly dispose of any unused solution. To avoid contamination solutions should not be put back into the stock bottle.

6. Broken glassware should be thrown away in a conventional trashcan.
   o False, put in the box marked broken glassware near the doorway.
   o True.

7. What should you do if a Bunsen Burner is burning and you smell gas.
   o Turn off the gas and notify your TA
   o Quickly notify your TA

8. Proper lab attire includes:
   o Open toed shoes, shorts, long baggy sleeves.
   o Closed toed shoes, pants, properly fitted shirts, long hair tied back out of your face and most importantly safety goggles.
9. If a chemical is spilled or splashed on hands
   o wash with copious amounts of water and report to your TA.
   o wipe it off with a paper towel and get back to work

10. If large quantities of hazardous chemicals are spilled or splashed on your body, you should
    o mop them up with a paper towel and get back to work
    o have someone notify a TA while washing with copious amounts of water under the safety shower.

11. If a chemical gets in your eyes,
    o rinse them in an eye wash for 15 minutes while holding your eyes open and have someone notify a TA
    o wipe them off with your sleeve

12. If a caustic substance such as an acid or a base is spilled, I should
    o wipe it up with paper towel and dispose of them in the contaminated paper waste receptacle
    o neutralize with sodium bicarbonate and wipe up with paper towels and dispose in the contaminated paper waste receptacle

13. Who is responsible for cleaning the common areas such as the hoods, balances and around the waste containers (solid, liquid, trash, broken glass ware and contaminated paper).
    o Me! and I realize that I will be penalized if I fail to assist in keeping the lab clean
    o My lab partners. That is what they are there for.
    o Nic or Kirk in the stockroom. They don't mind cleaning up after me.
    o My TA, they don't have anything better to do and surely a little mess left for them will make them happy with me while they grade my lab reports.

14. I understand that if I am in the lab without safety goggles and the proper attire while chemicals are being used, my actions may result in a failing mark for the term.
    a. A first offense will be dealt with by the TA (you will be asked to leave the lab until you have proper safety goggles and attire).
    b. A second offense will be require a meeting with Dr. Sheagley or Dr. Atkinson.
    c. A third offense will result in a grade of NP for the term.

    o Sign here to agree with the statement above: ______________________________

15. I understand the chemistry laboratory is an inherently dangerous place and I may be working with hazardous substances. I will do my best to actively understand the risks associated with each substance or technique used in the lab. I will act in a safe and professional manner.

    o Sign here to agree with the statement above: ______________________________
16. On this page, sketch the lab room. On your diagram, include the location of the safety shower, the eye wash, the fire blanket, fire extinguisher, fume hoods, contaminated paper waste receptacle, and broken glass receptacle. With each item, include a BRIEF description as to their purpose. Also note where the stockroom is in relation to your lab.
Week 1 Lab:

1. Check-in
2. Watch ACS safety video (link below)
   http://vimeo.com/6170550
3. Take safety quiz, turn it in to your TA
4. Do the Avogadro case study lab.
Introduction

The case study that follows was inspired by a novel assignment that Pace University Professor Carroll Zahn gave to his introductory computer class. The result of this assignment was certainly far different than anything that either the professor or his class could ever have predicted on the day that the assignment was given (Felsenthal, 1995).

One fateful day, Professor Zahn decided to challenge his class by asking them to calculate the cost of a single aluminum atom in a roll of aluminum foil that he had recently purchased. The information that he provided the class included the cost and size of the roll, the atomic mass of aluminum, and the value of Avogadro's number. After that they were on their own. Two students, Peter Broome and Marina Andre, became quite upset with the problem they were assigned, withdrew from the course, and, after failing to resolve their complaints to their satisfaction through standard academic channels, launched a lawsuit against Pace University. Their case was heard by New York State judge Thomas Dickerson who ruled in the students' favor, awarding them a full tuition refund for the course and $1,000 each in compensatory damages. The judge reportedly commented that, "Students are consumers—there is nothing holy or sacred about educational institutions." Pace University is reportedly appealing Judge Dickerson's decision.

At the risk of finding myself in court, Professor Schroeder, a fictional character in the case study that follows, is going to give you virtually the same assignment that Professor Zahn gave his class. A significant difference, however, is that you will be given the opportunity to design and perform simple laboratory experiments to obtain whatever information you deem necessary to solve the problem.

You are to work in groups to calculate the cost of a single aluminum atom in a roll of aluminum foil. As Professor Schroeder directs, your answer should be correct to three significant figures, should be documented with a detailed unit analysis, and should be reported using scientific notation. Use the cost information given in the case study. You will be given a piece of aluminum foil similar to that which our fictional Professor Schroeder gives to his students. Any experiments you may decide are necessary should be done using this piece of aluminum foil. All other necessary information is contained in your text.

Have fun, good luck, and, above all, let's work together to stay away from the legal system!
Avogadro Goes to Court—The Case Study

On a sunny Wednesday morning Professor Ken Schroeder entered his classroom and said, "Good morning, everyone. Isn't this a beautiful day?" Getting a minimal response from his students, he moved on. "I have decided to give you a different kind of assignment today, one that I hope you will enjoy and find instructive."

Dave Barton, a student who had been in another of Professor Schroeder's classes said, "I hope this isn't going to be another of your 'brain teasers,' Professor Schroeder."

"No, this is actually quite straightforward. I don't think that you'll have any trouble with it, and it should be interesting for you."

"It's the 'interesting' part that has us worried," said Kathleen Terry, a young woman who seldom spoke in class.

"Well, I assure you that you have nothing to worry about, Miss Terry. All that I shall ask you to do is to calculate the cost in dollars of a single aluminum atom in the roll of aluminum foil that I purchased today. Your answer should be correct to three significant figures and should be reported using scientific notation."

Dave Barton asked, "How are we supposed to do that?"

"I'll tell you the cost of the roll of aluminum foil and give you some additional information. The rest, including any measurements that you may wish to make, is up to you."

"The roll cost $1.59. The area of the roll as listed on the box is 6.96 square meters. I will give you an approximately 8 inch by 8 inch section of the roll that I have cut out for your use."

"That's all that we get?" asked a young woman in the back of the room.

"How much aluminum is in the roll?" asked a student in the third row.

"You should be able to determine that. Remember, Avogadro's number is central to your calculations."

"What's Avogadro's number?" asked another student.

Peter Brower asked, "Would you explain exactly what you mean by 'three significant figures'?"

"All of the information you need is either in your text or available to you if you make some measurements," said Professor Schroeder.

"You are to work on this as a group project outside of class. Start soon because your report is due one week from today. Be sure your report includes all of the details of your calculation, including a complete dimensional analysis."

"Professor Schroeder, some of us, at least, don't have a clue how to do this. We need some help," said Kathleen Terry.

"Just get started, Miss Terry. As you work together things will become much clearer to you."
From the nature of the conversations that ensued, it soon became clear that the class was confused and annoyed by this assignment. Professor Schroeder ignored this, however, and moved on to his lecture for that day.

At each of the next two classes the students peppered Professor Schroeder with questions about the aluminum atom problem. He consistently referred their questions back to them, telling them that they had access to all of the information that they needed, and that they had to state the problem properly in order to succeed. The students made little progress with the assignment, and became increasingly frustrated and angry as time went by.

One of the students suggested that they visit the Chemistry Department chairperson to complain about the lack of help that they were receiving from Professor Schroeder.

Professor Nancy O' Sullivan, the Chair of the Chemistry Department, met with the students later that same day. "Professor Schroeder has explained the nature of the assignment that he has given your class. It sounds reasonable to me, but I understand that you feel that you are being treated unfairly, is that right?"

"Yes," said Dave Barton, "we need help and Professor Schroeder won't answer our questions; he keeps telling us that we can find the answers in our text, and that we need to work together to solve the problem."

"Well, Professor Schroeder is right," said Dr. O'Sullivan. "All of the information is there for you. You need to work together, find it and get started on the problem. I'm afraid that I can't agree that Professor Schroeder is doing anything wrong. I'd advise you to get on with your assignment."

A subsequent visit to the Dean led to yet another rebuff and left the students frustrated and angry. After an additional attempt by a number of Professor Schroeder's students to petition the Dean led nowhere, the students angrily concluded that their only remaining recourse would be to the legal system. This seemed like a big step, but these are litigious times, and emboldened by a similar case that they had read about in the media, several of the students joined together to sue Professor Schroeder and his university.

When their case was heard in court, however, things went quite differently than the students had anticipated. The judge listened carefully and sympathetically to the arguments of both sides, but after some deliberation he ruled that the professor and his university were not at fault, and that the students did not have a valid case. He further ruled that the problem that Professor Schroeder had assigned was a good example of interactive group learning and a reasonable assignment.

Image Credit:

Portrait of Amedeo Avogadro
Edgar Fahs Smith Collection
University of Pennsylvania Library
Used with Permission

Date Posted: 08/10/99 caf. Last Revised 02/26/03 nas.
Avogadro Goes to Court  
CH 221 H/ CH 227H

A. Read the Case Study Handout (if haven’t already)

B. In your group discuss the following:

1. What differences are there between the fictional case and the real case?
   - List three differences.

2. In the introduction section, what are the students being asked to do?

3. On the first page of the case study (2nd page of handout), what questions indicate the students are confused or in need of more information?

4. What information do you want that is not given?

5. Where would you look for it?

6. Outline how to solve the problem. Give details of what measurements you will make. What tools would you need?

7. Discuss your outline with another group and come to a consensus that the measurements that both groups propose will lead to the desired answers.

C. Reconvene in the assigned lab rooms:

1. Decide how many experimental measurements you need to make before you are confident in your answer.

2. Complete the calculations necessary to report your value for the cost of a single atom of aluminum in the aluminum foil roll.
CH 221 H
Analyzing Currency
Pre-Lab Assignment

In the upcoming lab experiment (Case Study: Filthy Lucre) you will be analyzing US paper currency for traces of the chemical cocaine. The analytical technique we will be using is Gas Chromatography /Mass Spectrometry (GC/MS). It is unlikely that you have used an instrument like this before and we want to introduce you to the basic principles of the technique.

The chromatography portion of the technique might be familiar. This is used to separate compounds from one another. You may have done this to separate out colors in inks or dyes. To help you get familiar with the concepts in the technique, please view the you-tube video with the link below and answer the questions.

http://www.youtube.com/watch?v=4I9065A_6UY

1. Which ink color in the paper chromatography is most strongly attracted to the paper (stationary cellulose phase)?
2. What is Rf value and what is it used for?
3. In the column chromatography what is the initial mobile phase?
4. Why is a switch made to acetone as the mobile phase? *Hint: remember the old saying; like dissolves like.*

In addition to viewing the video and answering the questions, you should read the paper posted on D2L:

**Methamphetamine Contaminated Currency in the Birmingham, Alabama Metropolitan Area**
Brandon A. Fultz, Jessi A. Mann, and Elizabeth A. Gardner
*Microgram Journal, Volume 9, Number 2, pages 57-60.*

Use the questions below to focus your reading:

1. What chemical was detected on the currency?
2. What instrumental technique is used to detect the chemicals?
3. What information is being shown in figure 1?
4. Can you relate the Rf (retention time) to what you saw in the video?
5. What results are the authors reporting?

This paper is reporting a study very similar to what you will be doing in the lab experiment: Case Study: Filthy Lucre. In addition to this video and reading we will do an activity sheet on mass spectrometry during the down time in the lab.
Methamphetamine Contaminated Currency in the Birmingham, Alabama Metropolitan Area

Brandon A. Fultz, Jessi A. Mann, and Elizabeth A. Gardner*
Department of Justice Sciences
University of Alabama at Birmingham
1201 University Boulevard
Birmingham, AL 35294
[email: eagard@uab.edu]

ABSTRACT: GC-MS Analyses of extracts of one dollar bills collected from in and nearby the Birmingham, Alabama metropolitan area in 2012 indicated that 42% of them were contaminated with methamphetamine. This is the first time that methamphetamine was identified on one dollar bills since the laboratory began testing them in 2008.

KEYWORDS: methamphetamine, cocaine, currency, gas chromatography-mass spectroscopy, trace analysis, forensic chemistry.

Analysis of one dollar bills (USD1s) acquired in and near the Birmingham, Alabama metropolitan area for cocaine contamination has been an ongoing study at the University of Alabama at Birmingham Forensic Chemistry Laboratory since 2008. Most of the bills that have been analyzed were acquired in sets of 20 USD1s from local stores and banks. Since the program inception, between 40 and 85 percent of the bills in each set have tested positive for cocaine. Other reports of cocaine contamination of U.S. currency have given values ranging from 67 to 97 percent; however, most of these reports were for analyses of higher denomination bills [1-5].

In February 2012, a set of 20 USD1s was collected from a home improvement store in north Jefferson County, Alabama, about eight miles north of Birmingham (designated as NJC 1 in Table 1 and Figure 1). The results of the analysis of the 20 USD1s were unexpected. Only eight of the collected bills tested positive for cocaine, but 17 tested positive for methamphetamine. This was the first time that methamphetamine had been identified on a set of USD1s analyzed by this laboratory. In order to determine if these findings were a one-time occurrence or rather was indicative of a fundamental change in the drug contamination of bills in the Birmingham area, additional sets of 20 USD1s were obtained and analyzed, from: A) The same store in north Jefferson County (NJC 2); B) Downtown Birmingham (B’Ham); C) Bessemer (BES, about 15 miles southwest of Birmingham); and D) Grant (about 85 miles northwest of Birmingham). In addition, the chromatograms from the previously collected and analyzed bills (i.e., 2008 – 2011) were re-examined to determine if they had also been contaminated with methamphetamine, but not recognized as such at the time.

Experimental
The USD1 sets from north Jefferson County, Bessemer, and Grant were all collected from home improvement stores, whereas the downtown Birmingham set was collected from a fast food restaurant. At each location, all 20 bills were collected from a single cash register (our previous work with cocaine contaminated USD1s had shown that there were no differences in bills collected from a single versus separate registers; that is, there was no evidence of cross contamination from cashier handling or passive contact within an individual register). Each set of bills was placed in a zip-lock plastic bag by the respective cashier, and laboratory gloves were worn by the analysts who subsequently handled the bills. The serial numbers of the bills were recorded in the order they were analyzed (subsequently, the respective Federal Reserve Bank locations were recorded for additional data evaluation; however, because the date of issue does not necessarily correspond to the year the bill was printed, the dates of issue were not recorded).

Figure 1 - Gas chromatograms of extracts showing cocaine and/or methamphetamine from USD1s.
The extraction method developed by Negruz et al. was utilized [6]. Each bill was crumpled and placed into a 20 mL vial. Ten mL of 0.1 M HCl were added, and the vial was capped and agitated on an orbital shaker (150 cycles/min) for between 30 min and overnight. The resulting solution was transferred to a 20 mL vial, basified to pH 12 with 2 M NaOH, and extracted with 1 mL of CHCl₃.

The isolated extracts were analyzed using an Agilent 6890 gas chromatograph equipped with a DB-5 MS column (30 m × 0.25 mm, 0.25 μm film thickness), interfaced with an Agilent 5973 MSD. Helium was used as the carrier gas at a constant flow of 1 mL/min. The injector temperature was 260°C and the split ratio was 15:1. The GC temperature program started at 70°C (no hold), ramped at 20°C/min to 250°C (6 min hold).

Cocaine and methamphetamine standards were obtained from Sigma Aldrich. All other chemicals were chromatographic or HPLC grade. The retention time of the methamphetamine standard was 4.578 ± 0.05 min, and the fragment ions of m/z 58, 91, and 134 were used for confirmation. The retention time of the cocaine standard was 11.135 ± 0.06 min, and the fragment ions of m/z of 82, 182, and 303 were used for confirmation. Representative chromatograms are shown in Figure 1; in addition to methamphetamine and cocaine, several common contaminants are labeled, including a possible nicotine metabolite (5.73 min), acetyaminophen (6.04 min), diethyltoluamide (familiarly known as DEET, 7.16 min), diethyl phthalate (7.22 min), and 1,2-diphenoxethane (8.43 min). DEET is the most commonly used insect repellant, while diethyl phthalate and 1,2-diphenoxethane are used in production of various polymers. All of these contaminants were identified by comparison with the NIST mass spectra library [7] and were not confirmed. The chromatograms were not quantitated.

**Results and Discussion**

USD1s were originally chosen (in 2008) for this study both to reduce expense and because they are reportedly less likely to be contaminated with cocaine from trafficking versus any other denomination except for $100 bills [3]. Thus, in our opinion the results presented herein are more reflective of handling by cocaine and/or methamphetamine consumers, enabling local trends to be more easily identified and monitored.

The 2012 results are summarized in Table 1. In NJC 1, none of the 20 bills were positive for methamphetamine alone, none were positive for cocaine alone, and eight were co-contaminated with both cocaine and methamphetamine. In NJC 2, none were positive for methamphetamine alone, 13 were positive for cocaine alone, and five were co-contaminated. In B’Ham, none were positive for methamphetamine alone, 14 were positive for cocaine alone, and two were co-contaminated. In BES, three were positive for methamphetamine alone, six were positive for cocaine alone, and eight were co-contaminated. In Grant, three were positive for methamphetamine alone, seven were positive for cocaine alone, and four were co-contaminated. In total, 42% of the bills collected to date in 2012 tested positive for methamphetamine.

The chromatograms from previously conducted analyses (i.e., 2008 – 2011) were then re-examined to see if methamphetamine had actually been present on those bills but not recognized at the time. None of the chromatograms from any of the earlier analyses that were conducted using the conditions detailed in the Experimental section were positive for methamphetamine. However, one set of 10 bills collected in Birmingham in October, 2011 that had been analyzed using an alternate splitless GC method (for maximum sensitivity) did have three bills display ultra-trace-level peaks for methamphetamine. Based on these results, the contamination of USD1s with methamphetamine is a recent development in the Birmingham area.

Cocaine contamination of currency is international in scope, and has been thoroughly documented. There have been several mechanisms proposed for this contamination, including adsorption to the paper fibers, and absorption/dissolution in the various dyes that are imprinted on the paper and/or in the human sweat components and skin oils that become laced into the paper from normal handling of the bills. At the present time, the only identifiable trend is that most currency will test positive for cocaine in countries where cocaine abuse is widespread, and test negative in countries where such abuse is uncommon. According to Ebejer et al., it is currently not possible to correlate cocaine contamination with rural versus urban populations, percentage of convicted drug offenders in the area, proximity to a port of entry, geographical region, or socio-economic standing [8].

Contamination of currency with other drugs of abuse has been previously reported, although with less frequency and lower abundance versus cocaine. Heroin, morphine, O³-mono-acetylmorphine, methamphetamine, phencyclidine, tetrahydrocannabinol, and 3,4-methylenedioxyamphetamine have all been reported on currency [2,5,9-13].

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**Table 1 - Number and % of Drug Contaminated Currency.**

<table>
<thead>
<tr>
<th>Set</th>
<th>Number of Positive Samples</th>
<th>% Contaminated With Methamphetamine</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Methamphetamine Only</td>
<td>Cocaine Only</td>
</tr>
<tr>
<td>NJC 1</td>
<td>9</td>
<td>0</td>
</tr>
<tr>
<td>NCJ 2</td>
<td>0</td>
<td>13</td>
</tr>
<tr>
<td>B’Ham</td>
<td>0</td>
<td>14</td>
</tr>
<tr>
<td>BES</td>
<td>3</td>
<td>6</td>
</tr>
<tr>
<td>Grant</td>
<td>3</td>
<td>7</td>
</tr>
</tbody>
</table>
In 2001, Jenkins reported that 3 of 50 USD1s analyzed for drugs were positive for methamphetamine [2]; the bills in this study were collected from five different U.S. cities. Also in 2002, Nath et al. examined 80 bills from convenience stores in San Francisco and determined that methamphetamine could be detected on bank notes and that screening currency was indicated [11]. In 2010, Veitenheimer analyzed bundles of 1, 5, 10, 20, 50, and 100 dollar bills (10 bills per bundle) collected in 35 different cities for cocaine, codeine, heroin, MDMA, methamphetamine, and morphine. Nine of the bundles (4.3%) tested positive for methamphetamine [12]. And in 2011, Wimmer et al. identified methamphetamine on 53 of 64 Euros of mixed denominations; however, the average contamination per bill was only 7 ng, lower than cocaine (106 ng), benzoylecgonine (43 ng), heroin (41 ng), O\textsubscript{6}-monoacetyl-morphine (15.5 ng), morphine (16.5 ng), and MDMA (9 ng). Interestingly, all 64 of the Euros analyzed in this study were contaminated with cocaine [13].

As an additional measure of the randomness of the sample, the percentage of methamphetamine contaminated bills versus Federal Reserve Bank source was calculated for the Bessemer and Grant sets (Table 2). Although Birmingham is in the Atlanta Federal Reserve District, 11 of the 12 Federal Reserves were represented in the two selected sets; only the Minneapolis Federal Reserve was not encountered. Of the bills collected in BES and Grant, 27.5% were from the Atlanta Reserve Bank and 27.85% of the bills that tested positive for methamphetamine were from the Atlanta Reserve. Although the contribution from each individual Federal Reserve Bank is too small to assign any significance to them, collectively these values indicate that the location of issue is not a factor in the percentage of bills that are contaminated with methamphetamine.

<table>
<thead>
<tr>
<th>Federal Reserve Bank</th>
<th>Number of Bills</th>
<th>% of Bills</th>
<th>Methamphetamine Positives</th>
<th>Cocaine Positives</th>
<th>% Total Methamphetamine Contaminated</th>
</tr>
</thead>
<tbody>
<tr>
<td>Atlanta</td>
<td>11</td>
<td>27.5</td>
<td>5</td>
<td>8</td>
<td>27.8</td>
</tr>
<tr>
<td>Boston</td>
<td>3</td>
<td>7.5</td>
<td>0</td>
<td>1</td>
<td>0.0</td>
</tr>
<tr>
<td>Chicago</td>
<td>4</td>
<td>10</td>
<td>1</td>
<td>3</td>
<td>5.6</td>
</tr>
<tr>
<td>Cleveland</td>
<td>2</td>
<td>5</td>
<td>0</td>
<td>1</td>
<td>0.0</td>
</tr>
<tr>
<td>Dallas</td>
<td>2</td>
<td>5</td>
<td>2</td>
<td>2</td>
<td>11.1</td>
</tr>
<tr>
<td>Kansas City</td>
<td>1</td>
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<td>1</td>
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<td>5.6</td>
</tr>
<tr>
<td>Minneapolis</td>
<td>0</td>
<td>0</td>
<td>0</td>
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</tr>
<tr>
<td>New York</td>
<td>5</td>
<td>12.5</td>
<td>4</td>
<td>3</td>
<td>22.2</td>
</tr>
<tr>
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<td>1</td>
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<td>0.0</td>
</tr>
<tr>
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<td>16.7</td>
</tr>
<tr>
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<tr>
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Conclusions

Methamphetamine was detected on currency for the first time in the Birmingham metropolitan area. Forty-two percent of the bills collected to date in 2012 were contaminated with methamphetamine, more than has been previously reported for any drug other than cocaine in the United States. The high percentage of contamination detected in this study, and its sudden appearance, indicates a significant change in the pattern of drug contamination of currency around Birmingham, probably reflecting higher methamphetamine abuse in the local populace. This conclusion is in agreement with and complements the findings reported in the National Substance Abuse Index, which states that methamphetamine abuse currently exceeds that of cocaine throughout the state of Alabama [14].

By the time that contamination of currency with cocaine was detected, it was already widespread. The results of this study suggest that it is possible to track significant changes in methamphetamine abuse in a specific region over time. Future studies may lead to insights into the geographical and economic factors that influence methamphetamine abuse (if any). Determining the actual mechanisms for the absorption and/or adsorption of methamphetamine to currency is potentially an important area of future research.

References:


Lab Procedure – Chemicals in Paper Currency

Experimental Goals

1. Identify major chemicals and also the environmental (human health) relevant chemicals in the currency extracts
2. Determine concentrations of cocaine, triclosan, and THC in the paper currency extracts and spiking recoveries
3. Calculate cocaine, triclosan, and THC mass per paper currency bill

Materials Needed

1. Three (3) 40 mL vials
2. Three (3) autosampler vials with screw caps, labeled with the letter for your team
3. 20 mL of high purity methanol, in a 100 mL beaker (obtain from hood)
4. 2 of 1 mL and 1 of 5 mL volumetric pipettes and bulbs
5. 10 µL Syringes (2)
6. 50 µL Syringes (1)
7. One (1) clean test tube
8. Forceps
9. Internal standard solution (pyrene-d_{10} at 1000 ng/µL)
10. Surrogate standard solution (caffeine-d_{9} at 1000 ng/µL)
11. Cocaine solution at 1000 ng/µL
12. Triclosan solution at 1000 ng/µL
13. THC solution at 1000 ng/µL
14. Standard mixture 333 ng/µL (standard solution item 10, 11, and 12 are mixed in equal volume, stockroom will prepare this mixture)
15. Labeling tape and Sharpie Markers
16. One (1) US currency bill (denomination $1, $5, $10, or $20: higher the better since more likely to be contaminated with drug compounds)
17. **Teams H and higher only:** One (1) piece of printer paper cut to the size of a US currency bill or a brand new bill.

Note: Do not handle this with your hands. The point here is to analyze paper as a “blank”, so you can determine the levels of analytes as found in a “background” sample... i.e., paper that has not experienced all the handling that paper currency receives. The TA may give you some gloves with which to handle this paper.
Your TA will divide the class into two teams and assign a letter to each group in the team. Remember your group’s letter.

**Note:** Two different types of combination blank+spike recovery measurements will be carried out.
1. Half the teams will do a solvent blank + spike recovery test.
2. The other teams will do a solvent blank + paper + spike recovery test.
   *Your TA will determine who does what.

**All Teams: Sample Bill Extraction #1**

1. Practice taking up exactly 10 μL into a syringe, 1 mL, and 5 mL into pipettes. Ask your TA to check your technique before moving on.
2. Roll up the currency bill and place into the sample vial.
3. Go to the Surrogate Standard Station and add 10 μL of the surrogate standard (SS) solution, which is caffeine-d9, with a 10 μL syringe directly onto bill inside the sample vial.
4. Add exactly 5.0 mL of methanol to the sample vial using a clean volumetric pipette. Weigh the vial to within ±5 mg. This is Bill Weight #1.
5. Cap the sample vial with a cap that holds a Teflon lined septum. 
   Note: The rubber part of the septum should be towards the top, and the thin Teflon liner that is bonded to the septum should be towards inside of the vial (See Figure 1). **Place the vial in the sonication bath and sonicate for 1 minute. Remove and shake for 30 seconds.** Carry out the underlined/italics step a total of **three times.** (Sonication enhances the extraction by making sure there is fast transport of the analytes out of the sample and into the bulk solution of the solvent.
6. Go to the Internal Standard Station and add 10 μL of the internal standard (IS) solution, which is Pyrene-d_{10}, with a 10 μL syringe into each of the **three** auto-sampler vials and cap the vials (make sure the solution is spiked into the vial). (Target amount is 10 of the IS compound in each auto-sampler vial.
7. **Working quickly,** open the sample vial with the bill and carefully pour as much methanol as easily comes out into a clean test tube. (You will be leaving some methanol behind because the bill will stay wet.) Close the sample vial and set to the side for more work later. Without touching any surface of the auto-sampler vial, **quickly** pipette exactly 1.0 mL of the sample extract from the test tube into an auto-sampler vial (it contains the IS compound). Place a cap on the auto-sampler vial. Shake the vial to ensure that standards are well mixed. This is your **sample 1.** So if you are Team A, label it A-1.
   Note: The point of working quickly is so that little methanol evaporates and no concentration of the methanol occurs.

**All Teams: Sample Bill Extraction #2 (to see if you can get any more out of the bill by a 2^nd extraction)**

Fig 1
1. **Weigh the 40 mL sample vial from the first extraction of the bill.** This is Bill Weight #2. The volume of methanol you have remaining in the vial is:

\[
\text{Volume Remaining (mL)} = \text{Volume Originally Added (5.0 mL)} - \frac{\text{Bill Weight #1} - \text{Bill Weight #2}}{\text{density of methanol at room temperature}}
\]

Note: You need this volume because later it will let you calculate how much analyte you left in the vial just because some solvent from first extraction was left behind.

2. **Add 5.0 mL of methanol using a volumetric pipette to the sample vial containing the wet bill (do not allow the pipette to touch any vial surface).**

3. **Cap the sample vial.** Place the vial in the sonication bath and sonicate for 1 minute. Remove and shake for 30 seconds. Carry out the underlined/italics step a total of **three times**.

4. **Working quickly, open the sample vial and use a clean pipette to transfer 1 mL of the extract from the sample vial to an auto-sampler vial (it contains the IS compound).** Cap the auto-sampler vial and then shake the vial. This is your **sample 2**, so if you are Team A, label it A-2.

5. **Remove the bill using the forceps, place the bill on some lined bench paper in the hood and weight it down, allowing the methanol to evaporate.** The ventilation system may suck them up if they are not weighted down! Cap the sample vial and set it aside for collection, you will not be needing it anymore, but label it with your team letter.

**Teams A through ...: Combined Solvent Blank and Spike-Recovery Extraction**

1. **Go to the Surrogate Standard Station and add 10 µL of the surrogate standard (SS) solution, caffeine-d9, with a 10 µL syringe directly onto the wall of a new 40 mL sample vial.** This will be called your “solvent blank and spike recovery” (SB&SR) vial. Make a mental note where you apply the SS solution.

2. **Spike exactly 50.0 µL of the 333 µg/mL standard mixture solution, which contains three compounds likely found on currency: cocaine, THC (tetrahydrocannabinol) and the antimicrobial agent Triclosan, directly onto the other side of the wall of the SB&SR vial.** (This will give ~3 ng/µL in the final extract.)

3. **Add exactly 5.0 mL of methanol to the blank vial using a volumetric pipette without touching any SB&SR vial surface.**

4. **Cap the SB&SR vial with a cap that holds a Teflon lined septum.** Place the vial in the sonication bath and sonicate for 1 minute. Remove and shake for 30 seconds. Carry out the underlined/italics step a total of **three times**.

5. **Working quickly, open the SB&SR vial and use a clean pipette to transfer 1 mL of the extract from the vial to an auto-sampler vial (it contains the IS compound).** Cap the auto-sampler vial. This is your **sample 3**, so if you are Team A, label it A-3. Set the SB&SR aside for collection; you do not need it anymore, but label it with your team letter.

**Teams ... and Up: Combined Solvent + Paper Blank and Spike-Recovery Extraction**

1. **Using gloves provided, take a piece of the cut paper provided, roll it up, and place it into a new 40 mL vial.** This will be called your “solvent + paper blank and spike recovery” (S+PB&SR) vial.”
2. Go to the Surrogate Standard Station and add 10 µL of the surrogate standard (SS) solution, caffeine-d\textsubscript{9}, with a 10 µL syringe directly onto the paper blank inside the 40 mL sample vial.

3. Spike exactly 50.0 µL of the 333 µg/mL standard mixture solution, which contains three compounds likely found on currency: cocaine, THC (tetrahydrocannabinol) and the anti-microbial agent Triclosan, directly onto a different spot on the paper blank. (This will give \~3 ng/µL in the final extract.)

4. Add exactly 5.0 mL of methanol to the S+PB&SR vial using a volumetric pipette without touching the paper blank surface.

6. Cap the S+PB&SR vial with a cap that holds a Teflon lined septum. \textit{Place the vial in the sonication bath and sonicate for 1 minute. Remove and shake for 30 seconds.} Carry out the underlined/italics step a total of three times.

5. Working quickly, open the S+PB&SR vial with the paper and use a clean pipette to transfer 1 mL of the extract from the paper vial to an auto-sampler vial (it contains the IS compound). Cap the auto-sampler vial. This is your sample 3, so if you are Team H, label it H-\textbf{3}. Set the S+PB&SR vial aside for collection; you do not need it anymore, but label with your team letter.
Part I – Busted

Tom Brown raced through the airport to catch his plane. A Landscape Management major at Illinois A&M, Tom was returning to school after Christmas break. He was in a particularly good mood because Grandma Brown had given him $200 in cash as a Christmas present to spend “on something fun” at school. Tom had tucked the cash into his carry-on, which he held tightly in his hand. “I don’t want to leave this bag out of my sight,” he thought as he pondered ways he could spend the money.

He was suddenly jolted back to reality by a loud voice.

“Sir?”

“Yes,” replied Tom.

“Officer Adams, Drug Enforcement Agency. You’re under arrest. You have the right to remain silent....”

Tom didn’t hear the rest of the words. His mind was racing. “What’s going on here? What had he done?”

The voice of Officer Adams registered again. “Our drug sniffing dogs detected cocaine in your carry-on. You’re under arrest on suspicion of drug trafficking.”

“How could this be,” Tom thought, as Officer Adams led him into a nearby interrogation room. “I’ve never had anything to do with drugs.”

Background

This case study is based on an incident that happened in 1991. Willie Jones, a Nashville, Tennessee, landscaper, was arrested in an airport after dogs detected cocaine in his suitcase. Agents seized the suitcase, which contained $9,000 in cash. Under the provisions of the Racketeer Influenced and Corrupt Organizations (RICO) Act, the government kept the money. The government argued that since the money was contaminated with cocaine, it must have been used in the commission of a crime, namely drug trafficking. Mr. Jones later sued the government for the return of the money. His defense: cocaine contamination of money is now so widespread that detecting traces of it does not indicate it was used in drug trafficking.

Does Mr. Jones have a case (and, by extension, our friend Tom Brown)? Should the government return his money or not?
Questions

1. What is the Racketeer Influenced and Corrupt Organizations (RICO) Act?
2. Why did Congress enact RICO and what is its intent?
3. Do you think RICO was applied properly in Mr. Jones’s case? Why or why not? Can you find other instances where RICO was applied in a court case? Was RICO applied correctly in these cases? Why or why not?
4. What is cocaine? What is its chemical structure? How does cocaine affect the body?
5. How are dogs trained and used to detect drugs? How sensitive are their noses? What are some of the advantages and disadvantages of using dogs to “sniff out” drugs?
6. Mr. Jones’s defense in this case was that cocaine contamination is so widespread that the mere presence of cocaine on money is not indicative of criminal activity. How widespread is cocaine contamination in our money?
7. Put yourself in Mr. Jones’s defense attorney’s place. Design an experiment to determine what percentage of paper currency is contaminated with cocaine. Consider such things as: (1) sampling; (2) the method of analysis to use; (3) what is “contamination”; (4) what level of cocaine in money indicates criminal activity; and (5) how reliable are the results you might obtain from your experiment.
8. What is “sampling”? How do scientists obtain a representative sample from a group?
9. How does money become contaminated with cocaine in the first place?
10. If you were the judge in this case, how would you rule? Why?
Part II – Lab Work

If you have access to a gas chromatograph/mass spectrometer, you can perform the experiment(s) you designed in Part I.

Pre-Lab Work

Experimental methods exist to screen paper currency for the presence of cocaine (i.e., determine if cocaine is present in money or not). Before you begin any experimental work, however, compile a list of questions, or experimental variables, you should consider in order to help you decide if Mr. Jones does indeed have a case. Try to make the list as complete as possible. You do not have to know the answers to your questions, and some of your questions may not be answerable, but try to ask as many as you can. Here are three example questions to get you started in your thinking:

- How many paper bills are in circulation?
- How many do we need to test?
- How do we know we are measuring cocaine in our tests?

There are many more questions that we could ask about this problem. Try to generate as long a list as you can to add to these three.

Experimental Work

An instrument called a gas chromatograph/mass spectrometer (GC/MS, for short) is used to detect cocaine. You may not know what a GC/MS is, but you have probably heard of one. When athletes are tested for the use of banned performance-enhancing drugs, a GC/MS is the instrument used to do the tests.

Your instructor will tell you how much you must know about GC/MS. Here is a brief “big picture” overview of GC/MS for those of you who are not at all familiar with the technique. Gas chromatography (the “GC” part) is a powerful technique used to separate the components of a mixture. Mass spectrometry (the “MS” part) is then used to identify the components of the mixture. A urine sample from an athlete, for example, is injected into a GC. The GC then separates all the proteins, metabolites, drugs, etc., present in the urine into individual components. The individual components are then passed into the MS, which identifies what each component is.

In this project, paper currency will be treated with methanol to extract any cocaine present in the money. The extract will then be injected into the GC/MS, which will determine if any cocaine is present in the extract. Your instructor will tell you the maximum number of bills you can test. (The number will depend upon the size of the class, the time and equipment available, how much money you have, etc.) The procedure for extracting a bill is given below:

1. Roll the bill and place it into a clean vial.
2. Add 2 mL of methanol to the vial.
3. Cap the vial and shake for 1 minute.
4. Using a glass Pasteur pipette, transfer enough methanol to an autosampler vial to fill the vial about three-quarters full.
5. Remove the bill from the vial when you are finished using a forceps.
**Results**

After all the bills have been extracted, your instructor will run them on the GC/MS. Your instructor will explain how and where you can get the printout of the analysis of your bill. Be sure your class compiles a table of results for the entire class.

**Questions**

1. Based on the class results, what percentage of paper currency is contaminated with cocaine?
2. How confident are you that the class results represent all paper currency in circulation? Is there some way you could increase your level of confidence?
3. How many bills do you think you would have to test to get a representative sample of all bills in circulation? On what basis did you formulate your answer?
4. Did you observe any patterns to the contamination (for example, based on denomination or geography)?
5. What might you do in order to get quantitative results (i.e., determine how much cocaine was present in your bill)?
6. Based on the results of your experiments, if you were the judge in the Jones case, how would you rule? Why? Has your opinion of the case changed now that you have completed your experimental work?
7. Why is GC/MS the preferred method of choice for determining cocaine?
8. GC/MS is used to test athletes for banned performance-enhancing drugs. What are some of the advantages, disadvantages, and limitations to GC/MS for this purpose?


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<table>
<thead>
<tr>
<th>#</th>
<th>Compound</th>
<th>CAS #</th>
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<tr>
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<td>Glycerin</td>
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<tr>
<td>2</td>
<td>Urea</td>
<td>57-13-6</td>
<td>13</td>
</tr>
<tr>
<td>3</td>
<td>Phenol, p-tert-butyl-</td>
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<td>5</td>
<td>Dodecanoic acid</td>
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<td>mixture isomer</td>
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<td>Tetradecanoic acid</td>
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<td>Caffeine</td>
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<td>21</td>
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<tr>
<td>26</td>
<td>Oxiraneoctanoic acid, 3-octyl-, methyl ester, cis-</td>
<td>2566-91-8</td>
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<td><strong>THC</strong></td>
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<td>Squalene</td>
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<tr>
<td>30</td>
<td>Phthalic acid, bis(7-methyloctyl) ester and isomers</td>
<td></td>
<td>28--29</td>
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<tr>
<td>31</td>
<td>Cholesterol</td>
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<td>567-72-6</td>
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</table>

*Triclosan, Cocaine, and THC are in the calibration standard mixture.
(mainlib) Phthalic acid, bis(7-methyloctyl) ester
Naphthalene, 1-(phenylmethoxy)-
Diethyl Phthalate
1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester

(mainlib) 1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester
Scan 3282 (20.175 min): CC2013101101.D\data.ms (-3272) (-)

Caffeine-d9
surrogate standard
Cocaine
Caffeine
Dodecanoic acid
Benzene, 1,1'-[1,2-ethanediylbis(oxy)]bis-
n-Hexadecanoic acid
Nicotine

(mainlib) Pyridine, 2-(1-methyl-2-pyrrolidinyl)-
Pentadecanoic acid
Bis(2-ethylhexyl) phthalate
Dibutyl phthalate

(mainlib) Dibutyl phthalate
Tetradecanoic acid
Benzene, 1-(1,1-dimethylethyl)-4-(2-ethoxyethoxy)-
Phenol, p-tert-butyl-
Phenol, 4-(1,1,3,3-tetramethylbutyl)-
9,12-Octadecadienoic acid (Z,Z)-, methyl ester
9-Octadecenoic acid, methyl ester, (E)-
(mainlib) Dronabinol

27
Hexadecanoic acid, methyl ester
Oxiraneoctanoic acid, 3-octyl-, methyl ester, cis-
Pre-Lab: A Cycle of Copper Reactions

Part A

Answer the following questions in your lab notebook (be sure to show your work for any calculations):

1. In chapter 4 of your text, read about the different types of reactions. What causes a precipitate to form when certain combinations of aqueous salt solutions are mixed?

2. Locate the solubility table in chapter 4 and summarize which ions are generally soluble and which are generally insoluble.

3. Look up the MSDS for nitrogen dioxide gas. What dangers does it present and what steps need to be taken to avoid exposure?

4. This is one of the most dangerous experiments during this term because of the risk of exposure to dangerous chemical substances. For example, concentrated nitric acid is a particularly nasty solution; what happens when it comes in contact with skin? What safety precautions need to be taken to avoid exposure? All of the acids and bases used in this experiment are also potentially dangerous and should all be handled carefully.

Part B

Prepare your notebook for the lab. This includes stating the purpose of the experiment, summarizing the procedure in a bulleted-list format (be sure to include space for observations) and preparing any tables necessary for data collection.

At the start of your lab, remove the copies of the pages where you completed the above work from your lab notebook and turn them into your TA.
**A Cycle of Copper Reactions**

**GOALS:**

1. Cycle solid copper through a series of chemical forms via aqueous-phase reactions
2. Learn about and identify different types of aqueous reaction types
3. Calculate percent recovered copper after all of the transformations

**INTRODUCTION:**

This experiment will cycle elemental copper through a series of five reactions summarized below:

![Reaction Diagram]

The cycle will both begin and end with pure elemental copper. At different stages of the cycle, copper will be present in different chemical forms. At times copper will be present in solid compounds and other times in ionic form. Each chemical change that copper undergoes is observable as a change in the physical properties of the solution (or precipitate). As you perform each reaction, be certain to observe and record your observations of all physical changes.

At this point in the term, you should have been introduced (in the lecture class) to three different types of aqueous reactions: precipitation reactions, acid-base reactions, and oxidation-reduction (or redox) reactions (in addition, the text may have discussed gas-forming reactions). In precipitation reactions, soluble cations and anions combine to form an insoluble compound that leaves the solution as a solid precipitate. In acid-base reactions, an acid and base react to produce water and a salt. Redox reactions involve the transfer of electrons. As you go through the series of reactions you should be able to classify each reaction (with the exception of reaction 3) as one of the three above-described types of aqueous reactions.

**Reaction 1:** The first reaction proceeds according to the following **balanced chemical equation:**

\[
4 \text{HNO}_3 \text{(aq)} + \text{Cu} \text{(s)} \rightarrow \text{Cu(NO}_3)_2 \text{(aq)} + 2 \text{H}_2\text{O (l)} + 2 \text{NO}_2 \text{(g)}
\]
In this first reaction, elemental copper metal is reacted with concentrated aqueous nitric acid solution. The result of this reaction is that copper changes from its elemental state (charge = 0) to an aqueous, ionic state (Cu\(^{2+}\)) in an oxidation–reduction reaction.

**Reaction 2:** The second reaction then converts the aqueous Cu\(^{2+}\) into the solid copper (II) hydroxide (Cu(OH)\(_2\)) through a precipitation reaction with sodium hydroxide according to the following balanced chemical equation:

\[
\text{Cu(NO}_3\text{)}_2 \text{(aq) + 2 NaOH (aq)} \rightarrow \text{Cu(OH)}_2 \text{(s) + 2 NaNO}_3 \text{(aq)}
\]

**Reaction 3:** The third reaction takes advantage of the fact that Cu(OH)\(_2\) is thermally unstable. When heated, Cu(OH)\(_2\) decomposes (breaks down into smaller molecules) into copper (II) oxide and water according to the following decomposition reaction equation.

\[
\text{Cu(OH)}_2 \text{(s) + heat} \rightarrow \text{CuO (s) + H}_2\text{O (l)}
\]

**Reaction 4:** When solid CuO is reacted with sulfuric acid, the copper is returned to solution as an ion (Cu\(^{2+}\)) according to the following acid-base metathesis / double displacement reaction equation.

\[
\text{CuO (s) + H}_2\text{SO}_4 \text{(aq)} \rightarrow \text{CuSO}_4 \text{(aq) + H}_2\text{O (l)}
\]

**Reaction 5:** The cycle of reactions is completed in this reaction where elemental copper is regenerated according to the following oxidation-reduction reaction equation. This reaction changes copper from its ionic state (Cu\(^{2+}\)) to its elemental state by exchanging electrons between zinc and copper.

\[
\text{CuSO}_4 \text{(aq) + Zn (s)} \rightarrow \text{ZnSO}_4 \text{(aq) + Cu (s)}
\]

Here, zinc and copper exchange physical (and oxidation) states in and out of acidic solution. Hydrochloric acid is then used to dissolve any excess zinc. The solid copper can then be collected, washed, dried and weighed. Some copper is bound to be lost in all of the chemical transformations, so the percent recovery (mass copper remaining/initial mass x 100%) is expected to be less than 100%.
PROCEDURE:

Be sure to discard all waste in the waste jars as directed by your TA

Reaction 1:

Caution: Concentrated nitric acid is hazardous. Avoid getting it on your skin or clothing. If you do get any on your skin or clothing, wash it off immediately with running cold water. Do not breathe vapors.

Weigh out about 0.5 g of copper. Be sure to record the actual amount used to the nearest milligram. Place the copper at the bottom of a 250 mL Erlenmeyer flask. In a graduated cylinder, carefully measure out 5.0 mL of concentrated nitric acid.

DO THIS NEXT STEP IN THE HOOD!! NO₂ gas is toxic!

In the fume hood, add the nitric acid to the flask containing the copper. The nitric acid should completely cover the copper. Be sure to record all observations. Remaining in the hood, swirl the flask until all of the copper has dissolved. Once the reaction is complete and the gas has dissipated, add 20 mL of DI water to the flask. Once you are sure that all the gas has been removed in the fume hood, you may return to your workbench.

Reaction 2:

While stirring with a glass rod, slowly add 20 mL of 6.0 M NaOH to the flask. Be sure to record all observations.

Reaction 3:

With occasional stirring, slowly heat the flask on a hot plate until the solution just begins to boil. At this point you should notice that the blue Cu(OH)₂ has been converted to black CuO. If the conversion does not appear to be complete (not all of the blue Cu(OH)₂ has disappeared), heat the flask a little longer and/or ask your TA to take a look. Do not let the solution boil vigorously. Once the conversion is complete, remove the flask from the hot plate and allow the CuO to settle. In a clean beaker, heat ~ 200 mL of distilled water.

Add 50 mL of nearly boiling hot water to your reaction mixture. Once the CuO has resettled (give it about 5 minutes to settle), carefully decant the supernatant liquid, removing as much as possible without losing the desired product (CuO). Be sure to record all observations.
Reaction 4:

Carefully add 5 mL of 6.0 M H₂SO₄ (sulfuric acid) to the flask and swirl the mixture for 1 minute. All of the black CuO should dissolve and be gone at this point. If there is still black solid in your reaction mixture, add an additional 1 mL aliquot of the sulfuric acid and swirl the mixture for an additional minute. If the black solid has still not dissolved, ask your TA or repeat the addition of 1 mL of sulfuric acid as necessary. Be sure to record all observations.

Reaction 5:

**Caution:** Concentrated hydrochloric acid is hazardous. Avoid getting it on your skin or clothing. If you do get any on your skin or clothing, wash it off immediately with water. Do not breathe vapors.

**DO THIS NEXT STEP IN THE HOOD.** Hydrogen gas is generated which is extremely flammable. There should be no open flame in the room. Additionally, there is some possibility of producing more nitrogen dioxide gas, so do not breathe the vapors.

Add (all at once) 1.0 g of 30-mesh zinc or zinc powder. Stir until the supernatant liquid is colorless (not blue); the solution can be murky and grey. Decant the supernatant liquid. Remain in the hood and add 5 mL of distilled water followed by 10 mL of concentrated hydrochloric acid. The hydrochloric acid removes any excess zinc according to the following balanced chemical equation.

\[
\text{Zn (s)} + 2 \text{HCl (aq)} \rightarrow \text{ZnCl}_2 (\text{aq}) + \text{H}_2 (\text{g})
\]

If the hydrogen gas evolution stops before all of the solid zinc has been removed, more acid (in 1 mL aliquots) can be added. Once the evolution of hydrogen gas has become very slow, the flask may be returned to the workbench. You may warm the mixture on a hot plate to speed up the reaction, but do not boil the solution. Once the hydrogen gas evolution has completely stopped, remove the flask from heat and carefully decant the liquid. Transfer the solid copper to a clean pre-weighed beaker. Using a wash bottle to wash the copper metal into the dish can facilitate the transfer. Wash the copper at least twice with about 5 mL of distilled water each time. Decant the water after each wash. Wash the copper with an additional 5 mL of methanol. Allow the copper to settle and decant the methanol. Gently heat the copper on a hot plate to evaporate any remaining methanol and dry the copper. Once dry, remove the copper from the hot plate and allow the beaker to cool before determining the mass of the recovered copper. Be sure to record all observations and the final mass of the copper.
RESULTS:

Once the mass of recovered copper is known (difference between the pre-weighed beaker plus copper and beaker alone), the percent recovery can be calculated from the following formula:

\[
\text{Percent recovery} = \left( \frac{\text{mass of copper recovered}}{\text{initial mass of copper}} \right) \times 100\%
\]

REFERENCE:

Can we authenticate a painting?  
What tools and expertise can we use?

Preparation for Lab

1. Read for Friday
   - Art Under Scrutiny Case Study
   - One of the two articles below
       [http://pubs.acs.org/subscribe/journals/tcaw/11/i03/html/03lesney.html](http://pubs.acs.org/subscribe/journals/tcaw/11/i03/html/03lesney.html)
       [http://pubs.acs.org/cen/coverstory/7931/7931art.html](http://pubs.acs.org/cen/coverstory/7931/7931art.html)

2. Explore PHET Sim: Molecules and Light
   1. Record what you observed when IR light was incident on each of the molecules.
   2. Describe the motion of the atoms when IR light is observed.

3. Answer Pre-lab Questions
   You might need to read a couple of the references in the Case Study to answer these; I’ve highlighted some.

1. What is spectroscopy?
2. How can spectroscopy tell us anything about a painting?
3. What kind of information can you get from:
   a. UV Spectroscopy
   b. X-ray Fluorescence
   c. IR Spectroscopy
4. Why does UV light only penetrate 50 microns into the painting?
5. Why can IR be used to look beneath the painting?
Can we authenticate a painting?
What tools and expertise can we use?

Bellini Analysis:

Go to the website listed below and explore the research on Bellini’s “Feast of the Gods”

1. What information is available about this painting?
2. What techniques have been used to analyze it?
3. Can you determine the questions that they are trying to answer about the painting?
4. What conclusions do they come to?

URL: http://webexhibits.org/feast/analysis/xrayinfrared.html

IR Spectroscopy:

In the case study, the experts state that there are signals in the transmission IR spectrum that indicate particular functional groups that are characteristic of amino acids. Through work with molecular models, we will establish what this means.

Using the IPad app, ISpartan:

From: FC 03-1: Build water (H₂O), ethane (CH₃CH₃), ethanol (CH₃CH₂OH) and ethyl acetate (CH₃CH₂O₂CCH₃).

- Hints for building structures:
  1. When using the eraser, double click the region you want to delete.
  2. To replace a C atom in a ring with another atom, click the desired atom in the menu and then double click the atom you want to replace.
  3. The broom cleans up your structure, makes it look more like it “should”

After you build the molecules, click the search button at the bottom of the page.

1. Make sure the name it finds matches the molecule you are trying to build.
2. Click the 3D button
3. Click the download button at the top of the page – molecule with a red down arrow
4. Click the IR button at the top of the page.
5. Click on the spectra; Scan the spectra shown and stop on peaks. What motions do you observe? Record spectral numbers and describe motions.
Build the amino acids shown below and follow the same instructions above.

1. Identify peaks that can be attributed to the C=O stretch and NH stretch.

2. What do these peaks have in common?

3. What is meant by characteristic frequencies?

Three of the twenty common amino acids:

- proline
- alanine
- glycine

Final Analysis:

Referencing the Case Study on the Cezanne painting:

1. What is the evidence suggesting that the painting might be authentic?

2. What is the evidence suggesting that the painting is a fake?

3. In your group, come to a consensus and prepare to justify your decision to the class.
Analysis of Pigments

Pre-lab
Plan out your procedure and design a flow-chart to determine the identity of your unknown. Keep in mind you can observe both chemical and physical properties of the pigments. Read the XRF overview document. Then answer the two questions below.

1. What are the peaks seen in the XRF spectrum?
2. What is happening in the instrument and sample that gives rise to those peaks?

Art and artifact conservation relies upon analytical chemistry’s ability to discover what an object is made of in order to be able to design a proper treatment and conservation plan for the object. Today, conservation artists and scientists are often interested in the determination of authenticity of a painting, understanding the techniques used by the “old masters”, and determining the appropriate restoration or conservation treatment for a painting. These issues raise the following questions:

- What pigment did the painter use?
- Is there colored resin or varnish on top of the paint layer causing subtle alterations in the colors we perceive?
- Are we seeing the original color, or has the coloring material gone through alterations with time that have changed its appearance?

Identification of the pigments used in a painting can help us answer some of these questions and address the issues facing conservationists. Fortunately, pigment particles can be characterized by a number of physical properties as well as chemical behavior.

1. Color
2. Size and shape
3. Refractive index
4. Behavior under ultraviolet and/or polarized light
5. Reactivity with other chemicals

In order to carry out an appropriate conservation or restoration plan on a painting or painted object, it is first necessary to understand the composition of the paint. This means determining what type of binding agent was used in the paint preparation and identifying the pigments used as colorants. Pigment identification is also extremely important in painting authentication and dating because the presence of modern pigments such as titanium dioxide (TiO₂) which was not introduced until the 1920’s will immediately preclude claims of the painting being from earlier epochs. Pigments can usually be identified by a series of chemical and physical analyses. These analyses involve carefully looking at the pigment both macroscopically and microscopically, carrying out microchemical tests upon a sample of the pigment, and in some cases conducting various types of instrumental analysis on the pigment. However, most identification can be confirmed with microscopic viewing and a few selected chemical tests. In this part of today’s lab you will design an experiment to determine the identity of 5 white pigments and fillers.
Identity of Known Pigments
Chalk or whiting CaCO₃
Lead White 2 PbCO₃, Pb(OH)₂
Zinc White ZnO
Gypsum CaSO₄
Titanium White TiO₂

You will have known samples of all 5 pigments and you will have 1 unknown sample that is one of the five. The following reagents will be available to use in testing the pigments.

Reagents
3M HNO₃
KI crystals
3M HCl
10% H₂SO₄
1M BaCl₂
24 well plate

Use the following information to help you in designing your experiment and identifying your unknown.

a. Carbonates will dissolve with effervescence (bubbling) in acids.

b. Lead ion (Pb²⁺aq) will form a yellow precipitate of PbI₂ if mixed with KI.

c. Titanium white is an extremely inert pigment.

d. CaSO₄ will gradually form scattered acicular (needle-like) crystals upon standing in a dilute solution of nitric or hydrochloric acid. If no needle-like crystals are observed, a secondary test for sulfate ions should be performed. Using a pipette, draw off some of the acidified solution of CaSO₄. Adding BaCl₂ to that acidified solution will yield a precipitate.

e. ZnO will dissolve in acidic solutions, but not in water.

One method for carrying out chemical spot tests is to place small samples to be tested into a 24 well plate. Then you can add various solvents and reagents to the sample and observe it carefully. Once you have tested and observed all known substances, then you can carry out the same tests on your unknown sample. To confirm the unknown’s identity it is good to repeat the tests on the unknown and the suspected known in adjacent wells so that you can more closely compare the results.

Make a table with the sample number in one column and its likely identity as determined by the chemical spot tests and turn in the table to your TA – do this before proceeding to the final part of the lab: X-ray fluorescence analysis of pigments.

Waste Disposal
Collect all unknown waste and discard in provided container.

Discussion Questions
Write balanced equations for all reactions. Were any results surprising? Were the results obvious or are you unsure of the identity of some of the pigments based on the chemical spot tests? Describe the process by which you came to identify your unknown sample.

X-ray Fluorescence analysis of pigments
Your TA will bring samples of the five pigments to Dr. Clare’s research lab (SRTC-369) and you will be provided with XRF reference spectra via D2L. Your lab section will observe the data collection of each of your unknown pigments. The TA will distribute via D2L the XRF data collected.

Discussion Questions

Which is the major inorganic element present in each of the five spectra?
Make a new column in your table with the likely identity of each pigment as determined by XRF. Discuss any differences between the identity of the pigments by chemical spot tests and by XRF. Which method is more reliable? Explain your answer.
Overview of X-ray Fluorescence (XRF) spectroscopy

X-ray fluorescence (XRF) spectroscopy is an instrumental approach used to identify, and sometimes quantify, elements present in a substance. An element is identified by its characteristic X-ray emissions. The quantification occurs by measuring the intensity of the characteristic emission line(s).

As we have learned, all atoms have a fixed number of electrons arranged in orbitals around the nucleus, and the number of electrons in a given atom equals the number of protons in the nucleus. The number of protons is indicated by the atomic number in the Periodic Table of the elements. Each atomic number corresponds to a specific element. For example, the element tin has an atomic number of 50, and so the tin nucleus has 50 protons, with 50 electrons in orbitals about this nucleus.

Electron orbitals describe a specific distribution of electron density in space. As discussed in Chapter 6 of your textbook, orbitals with the same principal quantum number, n, are grouped together in an electron shell. Electron shells are then divided into sub-shells. Each shell is identified by an electron shell label, with the K shell corresponding to n=1, the L shell with n=2, etc. An important characteristic of electron orbitals is that each one corresponds to a specific and different energy level for a given element. This particular electron shell notation was first introduced by Charles Barkla, a physicist involved in the study of X-rays, who was awarded the Nobel Prize in Physics in 1913. The K shell includes electrons that are closest to the nucleus. As we shall see, XRF spectroscopy typically involves electrons in the K, L, and M electron shells. (Note: You should already have a good understanding of electron orbitals. The only thing “new” in this discussion is the terminology of K electron shells, L electron shells, M electron shells, etc. that are grouped according to the Principal Quantum number).

<table>
<thead>
<tr>
<th>Principal Quantum Number, n</th>
<th>Sub-shell designation</th>
<th>Electron Shell Label</th>
<th>Number of Electrons in Shell</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1s</td>
<td>K</td>
<td>2</td>
</tr>
<tr>
<td>2</td>
<td>2s, 2p</td>
<td>L</td>
<td>8</td>
</tr>
<tr>
<td>3</td>
<td>3s, 3s, 3p</td>
<td>M</td>
<td>18</td>
</tr>
<tr>
<td>4</td>
<td>4s, 4p, 4d, 4f</td>
<td>N</td>
<td>32</td>
</tr>
</tbody>
</table>

In XRF spectrometry, a high energy X-ray photon is emitted from an X-ray source and strikes the sample. High energy X-ray photons have sufficient energy to remove electrons from the innermost K or L orbitals. When this occurs the atoms become ions, since a negative charged electron has been removed. These ions are unstable. To return to a more stable electronic arrangement, electrons from an outer orbital, like L or M, move into the newly vacant inner orbital. In this process of an electron moving from an outer to an inner orbital an X-ray photon is emitted. This photon is termed a secondary X-ray photon, in contrast to the primary X-ray that was emitted from the X-ray source. This phenomenon of electrons moving from outer to inner orbitals with a corresponding emission of photons is called fluorescence. The
energy of the emitted fluorescent X-ray photon is dependent on the differences in energies between the initial and final orbitals of the individual transitions. Importantly, the secondary X-rays produced in this process are characteristic of a specific element. In other words, we can identify the elements present in a sample by analyzing the energy of the secondary X-rays.

![Figure 1](image1.png)

Figure 1. Depiction of an incoming X-ray photon promoting an inner electron to an excited state, thereby creating a “hole” near the nucleus. This “hole” is filled when an electron moves from a higher to lower energy level, and a secondary X-ray is emitted.

The most frequent transitions between outer and inner electron orbitals are given names. For example, an L shell to K shell transition is traditionally identified as a Kα (pronounced K alpha) transition. Other common transitions are shown in Figure 2. Remember, the order of electronic shells (beginning with closest to the nucleus) is K, L, M, N, etc. Each of these transitions will yield a fluorescence X-ray photon with a characteristic energy equal to the difference in energy between the outer (initial) and inner (final) orbitals. Using this terminology, what do you think a transition between an outer electron orbital of n=5 (O shell) and an inner orbital of n=4 (N shell) would be called?

![Figure 2](image2.png)

Figure 2. Depiction of electron orbitals and transitions that give rise to X-ray emission lines observed in X-ray fluorescence spectroscopy.

Inspection of Figure 2 reveals several features of X-ray emission lines, including 1) as the Principal Quantum number (n) increases, the electron orbital energies become
closer to each other, and so 2) the greatest energy difference between adjacent levels will always be between the L shell and the K shell; 3) when comparing the energy of K$\alpha$ and K$\beta$, or L$\alpha$ and L$\beta$ transitions, the $\beta$ transitions will always have a larger energy than the $\alpha$, since the $\beta$ are between non-adjacent energy levels. Combining these observations, one can conclude that the K$\beta$ transition will have the highest energy for a given element (revisit Figure 2 if this is unclear).

When analyzing a particular element by XRF spectroscopy, several of the transitions shown in Figure 2 will occur at the same time (with their respective characteristic energies) since many X-ray photons will be striking the sample. By sorting the energies of these photons a picture will be obtained that includes the intensity of radiation associated with the photons detected at each particular energy. This picture is the called a spectrum (plural is spectra). As defined in your textbook, a spectrum is the distribution among various wavelengths of the radiant energy emitted or absorbed by an object. Recalling that wavelength may be converted into energy, a spectrum displays the energies at which photons are absorbed, or in the case of fluorescence, emitted.

As an example of a spectrum, please see Figure 3 (top), which is an X-ray fluorescence spectrum of zinc nitrate. Also included in Figure 3 (bottom) is a spectrum obtained for a United States penny minted in 2008. This penny is a metal alloy made from Cu and Zn metals. Notice how the K$\alpha$ and K$\beta$ peaks for zinc occur at the same energies in Figure 3 (top and bottom), but at a different energy than the copper peak. This is the key to identifying elements by XRF spectroscopy! A given element will have an identifiable XRF spectrum, even if mixed with other elements, and each of these spectra are unique for each element. Table 2 contains the K$\alpha$, K$\beta$, L$\alpha$, and L$\beta$ emission line energies for selected elements. This information is used to identify elements in an X-ray fluorescence spectrum. (Note: For the penny in Figure 3, zinc’s K$\alpha$ and K$\beta$ peaks are identified, but only a K$\alpha$ peak is identified for Cu. Where is the Cu K$\beta$ peak in this spectrum?)

A constraint of X-ray fluorescence spectroscopy is that it is not generally suitable for analyzing light elements. The X-ray fluorescence spectrometer that we will be using will not be able to analyze elements with an atomic number below 19 (potassium). This may be viewed as a limitation, since our spectrometer is unable to identify common elements like aluminum or silicon in a sample. It is also unable to identify species like acetate, as demonstrated by comparing the results zinc acetate and zinc metal in Figure 3. However, a positive feature is that many sample holders that are made of organic polymers (primarily C, H, O) will be transparent for both primary and secondary X-ray photons. This means that the sample to be analyzed can be placed in a material transparent to X-rays, like a plastic bag, and analyzed directly.
Figure 3. X-ray fluorescence spectra obtained for polycrystalline zinc nitrate (top) and a U.S. penny (bottom), which is an alloy comprised of copper and zinc. The $K\alpha$ and $K\beta$ emission lines are shown.

It is apparent from figure that the $K\alpha$ line is the most prominent (highest intensity) for a given element. The reason for this is beyond the scope of our discussion. You may observe that the peak intensities (the $y$-values) are different for the Cu and Zn in the coin. This occurs because the intensity of each characteristic radiation peak is related to the amount of each element in a sample that is irradiated by the primary X-rays. This means that XRF spectroscopy may be used for both qualitative (identifying elements) and quantitative (determining the concentration of elements) measurements if the instrument is properly calibrated, i.e. the correlation between peak intensity and the concentration of the element has been established.
Table 2. K and L X-ray emission lines, in keV for selected elements.

<table>
<thead>
<tr>
<th>Atomic #</th>
<th>Element</th>
<th>Kα</th>
<th>Kβ</th>
<th>Lα</th>
<th>Lβ</th>
</tr>
</thead>
<tbody>
<tr>
<td>19</td>
<td>K</td>
<td>3.3138</td>
<td>3.5896</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>Ca</td>
<td>3.69168</td>
<td>4.0127</td>
<td>0.3413</td>
<td>0.3449</td>
</tr>
<tr>
<td>24</td>
<td>Cr</td>
<td>5.41472</td>
<td>5.94671</td>
<td>0.5728</td>
<td>0.5828</td>
</tr>
<tr>
<td>26</td>
<td>Fe</td>
<td>6.40384</td>
<td>7.05798</td>
<td>0.7050</td>
<td>0.7185</td>
</tr>
<tr>
<td>27</td>
<td>Co</td>
<td>6.93032</td>
<td>7.64943</td>
<td>0.7762</td>
<td>0.7914</td>
</tr>
<tr>
<td>28</td>
<td>Ni</td>
<td>7.47815</td>
<td>8.26466</td>
<td>0.8515</td>
<td>0.8688</td>
</tr>
<tr>
<td>29</td>
<td>Cu</td>
<td>8.04778</td>
<td>8.90529</td>
<td>0.9297</td>
<td>0.9498</td>
</tr>
<tr>
<td>30</td>
<td>Zn</td>
<td>8.63886</td>
<td>9.5720</td>
<td>1.0117</td>
<td>1.0347</td>
</tr>
<tr>
<td>35</td>
<td>Br</td>
<td>11.9242</td>
<td>13.2914</td>
<td>1.48043</td>
<td>1.52590</td>
</tr>
<tr>
<td>42</td>
<td>Mo</td>
<td>17.47934</td>
<td>19.6083</td>
<td>2.29316</td>
<td>2.39481</td>
</tr>
<tr>
<td>47</td>
<td>Ag</td>
<td>22.16292</td>
<td>24.9424</td>
<td>2.98431</td>
<td>3.15094</td>
</tr>
<tr>
<td>50</td>
<td>Sn</td>
<td>25.2713</td>
<td>28.4860</td>
<td>3.44398</td>
<td>3.6628</td>
</tr>
<tr>
<td>53</td>
<td>I</td>
<td>28.6120</td>
<td>32.2947</td>
<td>3.92604</td>
<td>4.22072</td>
</tr>
<tr>
<td>82</td>
<td>Pb</td>
<td>74.9694</td>
<td>84.936</td>
<td>10.5515</td>
<td>12.6137</td>
</tr>
<tr>
<td>83</td>
<td>Bi</td>
<td>77.1079</td>
<td>87.343</td>
<td>10.8388</td>
<td>13.0235</td>
</tr>
</tbody>
</table>

Prelab: Atomic Emission Spectra

Part A

Answer the following questions in your lab notebook (be sure to show work for any calculations):

1. What wavelengths of the electromagnetic spectrum correspond to visible light?

2. Why do atoms exhibit line spectra?

3. When light is emitted from the hydrogen atom, is the atom moving from a higher energy state to a lower energy state or a lower energy state to a higher energy state?

4. What is the equation for the energy levels of the hydrogen atom? Give the units associated with the energy equation you report.

Part B

Prepare your notebook for the lab. This includes stating the purpose of the experiment, summarizing the procedure in a bulleted-list format (be sure to include space for observations) and preparing any tables necessary for data collection.

At the start of your lab, remove the copies of the pages where you completed the above work from your lab notebook and turn them into your TA.
Atomic Emission Spectra Experiment

Goals
- To view the hydrogen emission spectrum and other atomic line spectra
- To contrast the line spectra with other broad-band sources
- To measure the wavelengths of the bright lines in the visible emission spectra of hydrogen and mercury, and calculate the energy of each line
- To gain an understanding of the quantized nature of the hydrogen atom

Supplies
- Simple transmission grating spectroscope
- Hydrogen and other elemental emission lamps
- White, red, and green light sources

Definitions
1. A spectroscope is an instrument that allows you to analyze light in some way. In this case, your spectroscope acts like a prism, splitting the light into different wavelengths.

2. A continuous spectrum is one in which a rainbow of colors is seen when viewed through a spectroscope or prism.

3. A line spectrum, when viewed through a spectroscope, appears as one or more sharp narrow lines (colors).

4. A band spectrum is intermediate in appearance – there will be a brightest color, but a range of nearby wavelengths are emitted.

Background
The understanding of the internal structure of the atom was advanced when Niels Bohr explained the cause of the emission spectra of atoms using the concept of quantization. He stated that electrons in the atom could exist at finite energy levels, but not in between them. Electrons within an atom can be excited to higher energy states through various means, including heating the atoms or using an electric discharge, like a spark. After exciting the electrons, the atoms emit electromagnetic radiation as the excited electrons relax into a lower energy state. The energy of the light emitted is equal to the difference between the energy levels in the atom. This emitted light can be passed through a prism or reflected from a diffraction grating to spatially separate it into its individual wavelength components (colors) generating an atomic emission spectrum, a line spectrum characteristic of the particular sample of atoms.

As an example, the emission spectrum of hydrogen consists of only four visible lines: red, blue-green, violet and deep violet (although your eyes may not be sensitive to the last one, since
individual’s visual acuity varies). Each color corresponds to the transition of an electron from an excited state, a higher principal energy level, to a lower principal energy level, possibly the ground state or some other allowed energy level in between. As the electron drops, it emits energy in the form of a photon which may or may not be in the visible region.

In this experiment you will use a spectroscope and gas discharge lamps to measure the wavelength of each bright line in the visible atomic emission spectra of both hydrogen and mercury. You will then use these measurements to calculate the photon energy for each bright line. Recall that the energy of light is related to the wavelength and frequency of the light, through Planck’s constant $h = 6.626 \times 10^{-34}$ J sec and the speed of light in a vacuum $c = 3.00 \times 10^8$ m/sec.

**Other Equations That Might Be Useful**

\[
\Delta E = - (2.178 \times 10^{-18} \text{ J}) \left( \frac{1}{n_{\text{final}}^2} - \frac{1}{n_{\text{initial}}^2} \right) \text{ [Rydberg equation, for Balmer series, } n_{\text{initial}} = 2]\]

\[
\Delta E = \frac{hc}{\lambda} \text{ [Links wavelength and energy – be careful of the units!]}\]

1 nm = $1 \times 10^{-9}$ m

*The final results of this experiment will be the wavelength (in meters) and the photon energy of each bright line measured for hydrogen and mercury.*
WARNING!!
The power supply for the discharge lamps operates at 5000 volts!
DO NOT TOUCH

How To Use The Spectroscope

Hold the eyepiece of the spectroscope up to your eye. Look through the slit at the small end pointing the widen end toward a fluorescent lamp (see figure 1, above). You will see the light through a vertical slit on the left and the spectrum of the light source projected onto a wavelength scale on the right. Adjust the position of the spectroscope until the light source, as seen through the slit, is as bright as possible. You will be revisiting this spectra later. First, note the graduations on the back of the spectroscope. The large numbers on the scale are hundreds of nanometers. See Figure 2 for how to read the scale. Each minor graduation represents 10 nm.

Figure 1 How a Spectroscope Works. A spectroscope is a small box, with a transparent grating in one side and a narrow slit directly opposite the grating. To observe a spectrum you point the slit toward a light source and look through the grating. You will see an image of the spectrum along the back wall of the box, just over the wavelength scale.

Figure 2: Observing a Spectra Using the Spectroscope. To determine the wavelength of the light you observe, you will make use of the calibrated wavelength scale which is in hundreds of nanometers.

One band of light that you should observe is a violet band the far right of the spectra. This band should be at 436 nm, note where that band appears on your spectroscope. A very rough correction factor can be made using the difference between your measurement and the accepted value (436 nm). If for instance you measured the violet band at 455 nm, your spectroscope is miscalibrated by about 19 nm. Any additional measurements made with this spectroscope should be corrected by subtracting 19 nm from the measurement.
**Spectrum of a Single Electron Element – Hydrogen**

- Record the line color and its position for the 3 or 4 brightest lines observed using the hydrogen lamp in table 1 and calculate each wavelength using Equation 1 from your lab manual. Be sure to include a sample of your work for one of the calculations.

**Table 1: Bright Line Spectra for Elemental Hydrogen.**

<table>
<thead>
<tr>
<th>Line Color</th>
<th>Spectral Line Position (nm)</th>
<th>Wavelength (nm) Corrected using calibration factor</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Spectrum of Multi-Electron Elements and Other Miscellaneous Spectra**

Qualitatively observe other atomic spectra and make notes below about the observed differences from the hydrogen spectrum.

Qualitatively compare the 3 brightest lines from the emission spectra of the elemental mercury lamp to the spectra of a fluorescent lamp and an incandescent bulb and the red and green colored sources provided.

Observe the solar spectra (if daylight is available).
Data Analysis
1) For the first four electronic transitions in the Balmer Series, calculate the change in energy of the electron \(\Delta E\), the predicted energy of the emitted photon \(E_{\text{photon}}\) and the predicted wavelength of the emitted photon \(\lambda_{\text{photon}}\). Put the calculated values in Table 2 and be sure to clearly show an example of each calculation in the space provided.

Table 2: Calculated Values for the Balmer Series of Hydrogen.

<table>
<thead>
<tr>
<th>Electronic Transition</th>
<th>(\Delta E) (J)</th>
<th>(E_{\text{photon}}) (J)</th>
<th>(\lambda_{\text{photon}}) (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(n_3 \rightarrow n_2)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(n_4 \rightarrow n_2)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(n_5 \rightarrow n_2)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(n_6 \rightarrow n_2)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Clearly show the following calculations for the \(n_3 \rightarrow n_2\) transition.

-- the change in energy of the electron, \(\Delta E\)

-- the predicted wavelength of the emitted photon, \(\lambda_{\text{photon}}\)

2) Based on your theoretical calculations, match the electronic transitions in the Balmer Series to the spectral lines you observed, and document your choices in Table 3. Then calculate the percent error between your experimentally determined and calculated wavelengths.

Table 3: Comparison of Experimental and Accepted Wavelengths from the Balmer Series.

<table>
<thead>
<tr>
<th>Spectral Line Color Observed</th>
<th>Experimental (\lambda) (nm) from Table 1</th>
<th>Accepted (\lambda) (nm) from Table 2</th>
<th>Electronic Transition</th>
<th>Percent Error</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>(n_3 \rightarrow n_2)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(n_4 \rightarrow n_2)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(n_5 \rightarrow n_2)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(n_6 \rightarrow n_2)</td>
<td></td>
</tr>
</tbody>
</table>

Below, clearly show your percent error calculation for the \(n_3 \rightarrow n_2\) transition.
3) It is not possible to observe the \( n_7 \rightarrow n_2 \) transition in the Balmer Series. Why do you think that is?

4) Emission spectra are sometimes referred to as atomic fingerprints. Is it possible to use them to identify elements in an unknown sample? Explain your reasoning: think about the Hg spectrum and that of a fluorescent bulb.

5) Why do you think sodium vapor lights cast a different color (yellowish) than fluorescent lamps?

6) Calculate the ionization energy of the hydrogen atom. Think about this process as taking an electron from its ground state, \( n = 1 \), to a position/energy level far, far away from the nucleus, \( n = \infty \).

Atomic Emission Spectra Lab Report:
There is not a formal lab report for this lab. Complete the above pages and submit them to your TA.
Determining the Concentration of a Solution: Beer’s Law

OBJECTIVES

In this experiment, you will

• Prepare beta carotene and curcumin standard solutions
• Use a Colorimeter to measure the absorbance value of each standard solution
• Find the relationship between absorbance and concentration of a solution
• Use the results of this experiment to determine the concentration of beta carotene and curcumin in your extracts

INTRODUCTION

The primary objective of this experiment is to determine the concentration of the natural dyes in your extracts. You will be using the Colorimeter shown in Figure 1. In this device, light from the LED light source will pass through the solution and strike a photocell. A higher concentration of the colored solution absorbs more light (and transmits less) than a solution of lower concentration. The Colorimeter monitors the light received by the photocell and reports either an absorbance or a percent transmittance value as compared to a blank, a solution containing no absorber.

You are to prepare five dye solutions of known concentration (standard solutions) and conduct a calibration procedure. Each standard solution is transferred to a small, rectangular cuvette that is placed into the Colorimeter. The amount of light that passes through the solution and strikes the photocell is used to compute the absorbance of each solution. When a calibration graph of absorbance vs. concentration is plotted for the standard solutions, a linear relationship should result, as shown in Figure 2. This linear relationship between absorbance and concentration for a solution is known as Beer’s law.

The concentration of beta carotene and curcumin in your extracts is then determined by measuring its absorbance in the same way with the Colorimeter. By locating the absorbance of the unknown solution on the vertical axis of the graph, the corresponding concentration can be found on the horizontal axis (follow the arrows in Figure 2). The concentration of the unknown can also be found using the slope of the Beer’s law line, assuming that the y-intercept of the calibration line is zero.
MATERIALS NEEDED

Vernier Colorimeter
one cuvette
one 5 mL pipet
pipet pump or pipet bulb
one 50 mL volumetric flask

PROCEDURE

1. Obtain and wear goggles

2. Obtain about 30 mL of beta carotene or curcumin stock solution in a 100 mL beaker. Add about 30 mL of distilled water to another 100 mL beaker. Be sure to record the concentration of the stock solution of beta carotene or curcumin from the container.

3. You will prepare five solutions of beta carotene or curcumin varying in concentration from approximately $6 \times 10^{-6}$ M to $2 \times 10^{-5}$ M. You may wish to check your concentrations and calculations with your TA before making the solutions. Make the solutions by pipetting the correct quantity of the beta carotene or curcumin stock solution into the volumetric flask and then filling to the line with distilled water. Be careful to avoid getting liquid above the fill line. Thoroughly mix each solution by inverting the stoppered flask ten times.

4. Connect the Colorimeter to the computer interface. Prepare the computer for data collection by opening the file “Lab 11: Beer’s Law” from the Chemistry 227 folder of LoggerPro. Set the colorimeter to a wavelength of 470 nm.

5. You are now ready to calibrate the Colorimeter. Prepare a blank by filling the cuvette 3/4 full with distilled water. To correctly use a Colorimeter cuvette, remember:

   - All cuvettes should be wiped clean and dry on the outside with a tissue.
   - Handle cuvettes only by the top edge of the ribbed sides.
   - All solutions should be free of bubbles.
   - Always position the cuvette with its reference mark facing toward the white reference mark at the top of the cuvette slot on the Colorimeter.

6. “Blank” the Colorimeter.

   Place the cuvette with the blank (water) in the colorimeter. In Logger Pro click on “Experiment”, then from the drop down menu select “calibrate” and “lab pro colorimeter”. In the popup window check the box next to “one point calibration”. Click “Calibrate now” and enter “100” in the box provided (100% T). Click “Keep” and then “Done”. You should now see an absorbance reading of 0.000.

7. You are now ready to collect absorbance data for the five standard solutions. Click [Collect]. Empty the water from the cuvette. Using standard solution 1 (the lowest concentration sample), rinse the cuvette twice with ~1 mL amounts and then fill it 3/4 full. Wipe the outside with a tissue and place it in the Colorimeter. After closing the lid, wait for the absorbance value displayed on the computer monitor to stabilize. Then click [Keep] and type the concentration of the standard solution into the edit box, and press the ENTER key. The data pair you just collected should now be plotted on the graph. [NOTE: When entering values, $2 \times 10^{-5}$ can be entered in standard
computer format as 2E-6.] You may need to click on the Autoscale button to rescale
the graph as you go along.

8. Discard the cuvette contents in the waste jar as directed by your TA. Rinse the cuvette
twice with standard solution 2 (next highest concentration), and fill the cuvette 3/4
full. Wipe the outside, place it in the Colorimeter, and close the lid. When the
absorbance value stabilizes, click [Keep], type the concentration of the standard
solution in the edit box, and press the ENTER key.

9. Repeat the Step 8 procedure to save and plot the absorbance and concentration values
of standard solutions 3-5. Wait until Step 12 to do the unknown. When you have
entered all of your standard solutions, click [Stop].

10. Be sure to record the absorbance and concentration data pairs that are displayed in the
table.

11. Examine the graph of absorbance vs. concentration. To see if the curve represents a
linear relationship between these two variables, click the Linear Fit button, . A
best-fit linear regression line will be shown for your five data points and your blank.
This line should pass near or through the data points and the origin (0,0) of the graph.
[Note: Another option is to choose Curve Fit from the Analyze menu, and then select
Proportional. The Proportional fit has a y-intercept value equal to 0; therefore, this
regression line will always pass through the origin of the graph.]

12. Put your prepared solution in a small clean beaker. Rinse the cuvette twice with the
unknown solution and fill it about 3/4 full. Wipe the outside of the cuvette, place it
into the Colorimeter, and close the lid. Read the absorbance value displayed in the
meter. (Important: The reading in the meter is live, so it is not necessary to click
[Collect] to read the absorbance value.) When the displayed absorbance value
stabilizes, record its value.

13. Discard the solutions in the waste jar as directed by your teacher.

**PROCESSING THE DATA**

You may use Microsoft Excel to plot the data and obtain a linear relationship between the
data, or you may use the following method:

1. Determine the unknown concentration: With the linear regression curve still
displayed on your graph, choose Interpolate from the Analyze menu. A vertical cursor
now appears on the graph. The cursor’s concentration and absorbance coordinates are
displayed in the floating box. Move the cursor along the regression line until the
absorbance value is approximately the same as the absorbance value you recorded in
Step 12. The corresponding concentration value is the concentration of the unknown
solution, in mol/L.

2. Print a graph of absorbance vs. concentration, with a regression line and interpolated
unknown concentration displayed. To keep the interpolated concentration value
displayed, move the cursor straight up the vertical cursor line until the tool bar is
reached. Enter your name(s) and the number of copies of the graph you want and
print.
3. Use the calibration curve equation to determine the concentration of beta carotene or curcumin in your prepared solutions (solve for Concentration, given Absorbance). **Calculate the concentration of beta carotene or curcumin in your solutions.**

4. Calculate the milligrams (mg) of dye you extracted from the plant material for each extraction.

5. Briefly describe your best extraction procedure, the one that yielded the most dye.

6. How do your best results compare to other groups results?

This lab was modified from lab 11 “Determining the Concentration of a Solution: Beer’s Law” from Chemistry with Computers, Third Edition, Vernier, Inc.