NJIT RET Summer program 2014
Lesson Module

MODULE TOPIC: Two Methods of Determining the Concentration of Soluble Compounds or Analytes.

LESSON ONE TOPIC: Colorimetric Analysis of Copper Ore

Learning Objectives
Students will be able to:
• Correlate the absorbance measured through the colorimetric analysis to the concentration of copper by plotting absorbance versus concentration.
• Determine the concentration of an unknown solution using a standard graph made by plotting absorbance versus concentration (M) using the Beer-Lambert law.

Exclusive Note to Teacher: Sample of powdered ore - A simulated copper ore can be made up with a minimum of 30% by mass of copper (II) carbonate, CuCO3(s), mixed with sand.

Standard (s)
NGSS: 5-PS1-3. Make observations and measurements to identify materials based on their properties.
CCSS-Math: 8.SP.2. Know that straight lines are widely used to model relationships between two quantitative variables.

Introduction:
An ore is mixture of a metal or a mineral with impurities or waste material. In order to find out if a mine is worth exploring, a sample of the ore is taken to a laboratory to determine the percent mineral of interest through colorimetric method. Colorimetric measurement of copper will be done using the Vernier colorimeter.

Figure 1 – Copper Ore.

Colorimeter is a device that is used to measure the absorbance of particular wavelength of light by a specific solution. This device is commonly used to measure the concentration
of a solute in a solution using the Beer-Lambert Law, which states that the concentration of a solute is directly proportional to its absorbance.

\[ A = -\log T = \varepsilon bc \quad \text{Beer-Lambert Law} \]

A= Absorbance  
b = The path length through the sample  
c = concentration

\[ \varepsilon = \text{Proportionality Constant} \]

Figure 2 - A simplified diagram of a colorimeter  
http://www.docbrown.info/page07/SSquestions/colorimeter.gif

Prior to determining the concentration of the mineral, a standard graph must be generated. This graph indicates the relationship between the absorbance and concentration of a series of known solution (standards) as shown in the figure above.

To measure the amount of absorbance of a particular wavelength of light, the sample is poured into a cuvette (shown below).

Figure 3- Cuvette used with the colorimeter. 
http://www.capitolscientific.com/core/media/media.nl?id=54179&c=1250437&h=9ea46b90a594c5b0dc28
The handling of the cuvettes is extremely important. Any stains, smudges, or scratches will cause varying results. Thus, it is essential to follow several rules in dealing with cuvettes:

1. Do not handle the lower portion of a cuvette through which the light beam will pass.
   a. Beaker (250 cm$^3$)
   b. Beaker (100 cm$^3$)
   c. Volumetric flask (100 cm$^3$)
   d. Volumetric flask (50 cm$^3$)
   e. Small filter funnel and filter paper, to fit volumetric flask
   f. Plastic weighing dish (boat)
   g. Measuring cylinder (50 cm$^3$)
   h. Measuring cylinder (10 cm$^3$)
   i. Access to a balance (weighing to the nearest 0.1 g)
   j. Computer with Vernier logger pro software
   k. Vernier Colorimeter
   l. Micropipette, 1-5 ml

2. Always rinse the cuvette with several portions of the solution before taking a measurement.

3. Wipe off any liquid drops or smudges on the lower half of the cuvette with a clean Kimwipe or other lens paper before placing the cuvette in the instrument. Never wipe the cuvette with paper towels or handkerchiefs. Inspect the cuvette to ensure that no bubbles are clinging to the inside walls. If you observe any trapped air bubble, just tap it gently to push them up into the air.

4. When inserting a cuvette into the sample holder:
   a. To avoid any possible scratching of the cuvette in the optical path, insert the cuvette with the index line facing toward the front of the instrument.
   b. After the cuvette is seated, line up the index lines exactly.

BACKGROUND INFORMATION:
- Students have studied the electromagnetic radiation and the relationship between speed of light, frequency and wavelength.
- Students have received a lesson on Beer-Lambert Law.
- Students know the unit of solution concentration in Molarity.

Health & Safety Rule:
1. Wear eye protection throughout.
2. Wear gloves, dilute sulfuric acid, H$_2$SO$_4$ (aq) is corrosive.

Chemicals:
- Purified (demonized or distilled) water
- Dilute sulfuric acid, approx. 2 M (CORROSIVE), 40 cm$^3$
Sample of powdered ore (see technical notes) (HARMFUL, DANGEROUS FOR THE ENVIRONMENT), 10 g
Copper (II) sulfate solution, 1 M, 25 cm³ (HARMFUL, DANGEROUS FOR THE ENVIRONMENT)

Apparatus:
- Eye protection
- Each student or pair of students will require:
  - Access to a printer to print the graph

Procedures:
Part I – Standard Solution Preparation and Measurement
1. Using the copper (II) sulfate solution provided, prepare six 50 ml solution of diluted copper (II) sulfate using 50 ml volumetric flasks, according to the following table. Ensure the solutions are well mixed. Calculate their concentrations in Molarity and record them in the table below.

<table>
<thead>
<tr>
<th>Volumetric Flask #</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume of copper (II) sulfate solution (mL)</td>
<td>30</td>
<td>24</td>
<td>20</td>
<td>16</td>
<td>10</td>
</tr>
<tr>
<td>Volume of purified water (mL)</td>
<td>20</td>
<td>26</td>
<td>30</td>
<td>34</td>
<td>40</td>
</tr>
<tr>
<td>Calculated Concentration (M)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Observed Absorbance</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- Table of Standard Solutions.

2. Attach the Vernier colorimeter to the computer, open up the logger pro software and set the wavelength to 620 or 630 nm and let the colorimeter warm up for 15 minutes prior to any measurement.

3. Rinse out a cuvette with deionized (DI) water and fill it 2/3 with DI water. This will be your “blank” solution. Wipe off the outside of the cuvette with a kimwipe, and place it in the spectrophotometer. Close the sample cover. Follow the steps to “zero” the spectrophotometer. Water does not absorb light in the visible region of the electromagnetic spectrum, so it should measure zero absorbance. You will adjust the instrument so that it actually does read zero absorbance with the water in the sample chamber.

4. Dump out the water from the cuvette, then rinse it thoroughly three times with small portions of the standard copper solution that you made. Each time, discard the rinse solution into a waste beaker. The purpose of this step is to make sure that you don’t dilute the concentration of the solution you made with any drops of water still
adhering to the inside of the cuvette. This way, you can be sure that the solution you are measuring the absorbance of is still the same concentration as the solution you made. To summarize the steps: rinse the cuvette three times with the first solution, fill it with the solution, wipe off the outside of the cuvette with a kimwipe, and then measure the absorbance of this solution.

5. Repeat this procedure for each solution to be tested and record the absorbance in the table above.

Note: Rinse the cuvette three times with the same solution prior to each measurement.

Part II - Preparation of the Ore or unknown sample
1. Weigh out as exactly as possible 10 g of the ground ore and transfer it into a 250 cm³ volumetric flask.
2. Add 40 cm³ of the dilute sulfuric acid a little at a time, allowing the effervescence to subside between additions.
3. When the reaction has finished filter the mixture into the volumetric flask.
4. Add purified water until the total volume of liquid in the flask is exactly 100 cm³.
5. Measure the absorbance of the unknown solution using the logger pro software at 620 or 630 nm.

Result & Data Analysis:
1. Calculate the concentration (in M) of the standard copper solution you made in Part 1.
2. Calculate the concentration of copper ions for the unknown in Part 2.
3. Using the logger Pro software make a graph of absorbance at 620 nm vs. concentration of copper (II) ions in units of Molarity. You will have 5 points, plus the origin (0,0). Since the absorbance of a solution containing no copper ions should be zero, (0,0) is one of the points to plot. Follow the graphing guidelines. Draw the best straight line among the points.
4. Using the straight-line equation from the graph, determine the concentration of copper ions in your unknown solution from its absorbance at 620 nm.
5. From the concentration of your unknown and the volume of the solution, determine the number of moles of copper present in the unknown sample. Don’t forget to take into account any dilutions you made.
6. Determine the mass of CuSO₄•5H₂O present in your unknown sample. Calculate the percent purity of the sample. (% Purity = mass of compound present / mass of entire sample x 100).
7. When done, report you calculated value and ask your teacher for the actual % Purity to calculate your percent error.
8. Answer the following questions and hand in your typed report in one week.

Post Laboratory Questions: (20 Points)
1. (2 Points) When preparing the standard solution in Part 1, why can’t we just put the solid in the flask, fill it with water up to the mark, and then mix.
2. (2 Points) Why is it necessary to rinse out the cuvette with the solution to be used in it before making our measurements?

3. (2 Points) Why must we use the same cuvette for all measurements?

4. (2 Points) What is the purpose of “zeroing” the spectrophotometer using a “blank” solution (water)?

5. (2 Points) What assumptions are we making in the calculations and analysis for this lab?

6. (2 Points) Prepare a graph showing the relationship between concentration and absorbance for a solution.

7. (8 Points) 6.019 g of an unknown copper compound was dissolved in 100.0 mL solution. The absorbance of this solution was 0.477 at 620 nm. Use your Beer’s law graph to determine the concentration of copper ions in this solution. Then determine the mass percent copper in the copper compound.

Grading:
1. Follow the given rubric to write your report. (70 Points)
2. Post laboratory questions (20 Points)
3. % Error (10 Points)
   Point deducted based on percent error:
   1. Percent error 0-5% = 0
   2. Percent error 6-11% = 3
   3. Percent error 12-20% = 5
   4. Percent error 21-25 = 8
   5. Percent error > 26 = 10

Assessment:
• Students calculate the concentration (in M) of the standard copper solution.
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- Students **plot graphs** (scatter plot) of absorbance (A) vs. Concentration (M) of the standards solutions of CuSO$_4$•5H$_2$O using logger pro software.
- Students **interpolate the concentration** of the unknown solution of copper ore dissolved in sulfuric acid using the **linear fit of the scattered graphs**.
- Students **calculate the Percent purity** of CuSO$_4$•5H$_2$O in the ore using the actual given value.
- Students **answer the post laboratory questions**.
- Students **write a formal lab** report following the provided rubric incorporating all the above items.

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Contributors
Mina Armani, Union City High School, Union City, NJ, Primary Author
Howard Kimmel, Levelle Burr-Alexander, John Carpinelli - Center for pre-College Programs, NJIT.
Dr. M. Azad, Dr. B. Patel, Dr. R. Dave - C-SOPS, NJIT
LABORATORY REPORT FORMAT (40 points)

Laboratory reports should follow this format unless announced otherwise. It is important to recognize that when you are writing a lab report, you should be objective. Also, the lab reports are used as examples of your writing style, so when sentences are written, they must be grammatically correct. You should use a computer; use Times New Roman font size 12, double-spaced. If the calculations section of the lab report is handwritten, it should be done neatly in pencil (erase errors), on lined paper, one side only.

Lab reports are due at the beginning of the class on the due date. If you are missing class (for example, a field trip) the report should be in my mailbox before you leave. If you are absent the date it is due, you must hand it in before homeroom the day you return. Penalties: minus 1 grade per day. Lab reports are usually 40 points each.

Lab reports are collected and kept on file; for AP students, a small folder or binder is appropriate, since some colleges require a summary of the experiments done as well as the AP Test score; the collection will be returned to you when you graduate.

FORMAT:

I. Title Page  title in the center of the page (2 points)
   Your name, date performed, class period, and partner(s)
   Abstract (should take up about 1/3 of the bottom of the page) (5 points)
   • a brief summary of a research article, thesis, laboratory report or any in-depth analysis of a particular subject
   • placed prior to the introduction and procedure (on cover page of lab report)
   • often used to help the reader quickly ascertain the paper’s purpose

II. Copy of the experiment with directions (3 points)
    This includes the Objective, Procedure, Theory, etc.
    If these are not included (or stained), you need to write them yourself

III. Data and Results (5 points)
    Even if you scribbled the data on your lab directions, you need to create the appropriate tables, graphs, descriptions, etc. (use your computer)

IV. Calculations (10 points)
    Show all calculations that you used to get your results. Be sure to use correct significant figures and labels. If you are not using Equation Editor, the handwritten page should be done in pencil on lined paper.
    If lab is qualitative and there are no calculations, points can be adjusted.

V. Conclusion (10 points)
   This section includes:
   ANSWERS TO QUESTIONS given in the lab exercise.
   • Write question in italics
   • Write the answer below it.
   FINAL PARAGRAPH:
   • Your answers to the objective of the experiment (the result of
   • The experiment, not what you should have gotten
   • An explanation for all large experimental errors (in excess of 10%)
   • Any error greater than 30% calls for repeating the experiment