Lab 3: Trends in the Copper Content of Pennies from 1983 to 1992

Week 1 prelab

1. Look up the mass of copper in a modern penny. Calculate the mass of Cu in the penny if the penny is 2.5% Cu by mass.
   http://www.usmint.gov/about_the_mint/?action=coin_specifications

Mass penny (g)	
Mass Cu (g)	

2. Calculate the concentration, in ppm, of copper in solution if you take a penny (mass Cu determined in question 1) and dissolve it in 100 ml.

Cu (ppm)	

3. What volume of the solution made in question 2 must be diluted to 100 ml so that the resulting final solution is 2.5 ppm Cu.

Cu (ppm)	Volume (ml)
2.5	

4. Calculate the volumes of 100 ppm Cu required to prepare 100ml of 1, 2, 3, 4, and 5 ppm Cu standards.

Cu ppm	Volume (ml)
1	
2	
3	
4	
5	

5. Calculate the concentration, in ppm, of Cu in solution if you dissolve of 50 mg Cu in 100 ml. What volume of that solution would be required to make 100 ml of 5 ppm Cu solution.

[Cu] Soln 1 (ppm)	
Vol soln 1 in soln 2	

Lab 3: Trends in the Copper Content of Pennies from 1983 to 1992

Before lab (week 1):

1. Do prelab worksheet
2. Prepare your lab notebook and data tables
3. Examine MSDS sheets on website: $HNO_{3(aq)}$, $NO_{(g)}$, $NO_{2(g)}$

Week 2:

1. Determine order in which you want to run samples. Write it down.

Week 3:

1. Begin calculations
2. Bring your laptop if you prefer to work on it.

Introduction

Success in modern manufacturing can only be achieved if the manufacturing processes have high precision and high accuracy. In practice, this means that virtually all of the produced materials must achieve the desired specifications so that there are few failures. In addition, there should be a little variation in the quality of the produced materials. Both goals must be achieved in the context of minimizing manufacturing costs.

Even the production of a simple object like a penny requires manufacturing to very high tolerance standards. The modern penny has a zinc core and a thin copper shell. The shell must be thick enough to resist abrasion that would expose the zinc core to corrosion. However, a shell that is too thick raises the cost of the penny to an undesirable extent. The US Mint did preliminary experiments and determined that the optimum thickness of the copper shell corresponded to a composition of roughly 2.5% by weight copper.

In 1982, the U.S. Mint began production of the copper clad, zinc-cored penny. It replaced a penny that was an alloy of 95% Cu and 5% Zn. It is a reasonable hypothesis that the thin copper shell was thicker and more variable during early years of production than it was during later years. You are going to sample the first ten-year history of pennies and determine if the copper content of pennies varied at a measurable rate. That is, you are going to determine if there is a significant *correlation* between year of mining and copper content. There are two possible sources for a systematic change in the mass of copper: 1) A change in the thickness of the copper shell, or 2) the zinc-core was contaminated with copper early in the manufacturing cycle and the degree of contamination could have decreased with time.

You will also determine the major source of variance in the study that you make. It could be due to instrument measurement variation, variation introduced in the bench chemical operations, or variation among the penny samples. Identification of major sources of variance is important in iterative investigations so that subsequent efforts to improve the precision can be focused on the correct part of the overall procedure.

The overall goal of this experiment is to determine if the thickness of the copper shell of pennies decreased regularly from 1983 to 1992 due to an increase in manufacturing capabilities.

Safety

MSDS sheets are available on the website for the following materials: nitric acid, nitrogen oxides (NO, NO_2). Review them prior to beginning the experiment.

Concentrated nitric acid (70% or 16 M HNO_3) and 6 M HNO_3 are strong oxidizing agents. If mixed with organic material, it reacts explosively. Nitric acid is a severe poison if ingested. Nitric acid will cause burns if it is in contact with the skin for more than a few seconds. You must wear rubber gloves and goggles when you are pouring these nitric acid solutions. When you pour these solutions, the vessels must be in the hood with the sash down and your eyes should be well above the level of the flowing stream of liquid. You can make close visual checks of volume delivered after you have placed the nitric acid back on the hood bench top.

The oxides of nitrogen (brown gas) formed when copper reacts with nitric acid are suffocating and hazardous.

The flame in the atomic absorption spectrometer is made by burning a mixture of acetylene and air. It is hot, approximately 2500K. The burner on the atomic absorbance spectrometer remains hot after the flame is extinguished.

Procedure

Week 1: preparing Cu standards and samples

1. Acid wash 100 ml volumetric flasks (quantity 26)

The 0.1 M hydrogen ions will be able to displace the small amount of Cu^{2+} that might be sorbed onto cation exchange sites on the surface of the glass.

These flasks are only to be used for solutions containing less than 10 ppm copper.

a. Rinse each flask with two portions of RO water.

Italics steps to be performed in the hood while wearing gloves

b. Fill each flask to the top with 0.1 M nitric acid ("acid wash 0.1 M nitric acid") and soak for 15 minutes.

c. Pour the nitric acid back into the bottle that it came from. It is reusable.

d. Rinse each flask with 4 or 5 small portions of RO water.

2. Dissolve 10 pennies (takes 20-40 minutes to dissolve)

a. Get a penny manufactured in each year from 1983-1992

b. Weigh each penny on the 4-decimal balance

Italics steps to be performed in the hood while wearing gloves

c. Dissolve each penny in 7.5 ml of 16M HNO_3 in a 100 ml beaker

d. React 15 minutes

e. Label your volumetric flasks (not acid washed) while the pennies are dissolving with the year of the penny
f. Record the mass of each volumetric flask to 3 decimal places
g. If penny is not dissolved, add another 2.5 ml of 16 M HNO_3
h. When pennies are completely dissolved, quantitatively transfer solution to a labeled 100 ml volumetric flask and dilute to 100 mL
i. Put a cap on and stir by inverting 12 times
j. Record the mass of each full volumetric flask to 3 decimal places

3. Dilute penny solutions to ~2.5 ppm Cu for analysis

This is the step most likely to mess up your results, so be careful. Maybe calibrate your pipet on the balance with water before beginning the dilution step.

a. Label acid washed 100 ml volumetric flasks with penny years- make duplicate flasks for 2 of your penny solutions to assess precision of bench-top techniques
b. Record the mass of each empty volumetric flask to 3 decimal places
c. Do <u>duplicate dilutions of two penny samples</u> to assess precision of bench-top techniques
d. Dilute penny solutions to ~2.5 ppm according to your prelab calculations
e. Record the mass of each full volumetric flask to 3 decimal places
f. Put a cap on and stir by inverting 12 times

4. Make standards from 100 ppm Cu stock solution

a. Take a 20 ml aliquot of stock solution to your bench
b. Record the exact concentration of the stock solution
c. Label acid washed 100 ml volumetric flasks 0, 1, 2, 3, 4, 5, and 6 ppm Cu standards
d. Record the mass of each empty volumetric flask to 3 decimal places
e. Generate standards according to your calculations in 100 ml volumetric flasks.
f. Record the mass of each full volumetric flask to 3 decimal places
g. Put a cap on and stir by inverting 12 times

5. Preparation of individual unknown standards

a. Get an unknown solid from the TA- be sure to record your name on the unknown sheet next to the unknown you grab. Record the number of your unknown in your lab notebook. These unknowns contain 10-50 mg Cu.
b. Record the mass of each labeled, empty volumetric flask to 3 decimal places
c. Quantitatively transfer your unknown to a 100 ml volumetric flask and dilute to volume.
d. Record the mass of each full volumetric flask to 3 decimal places
e. Put a cap on and stir by inverting 12 times
f. Perform <u>duplicate dilutions</u> to ~5 ppm Cu according to your prelab calculations. Be sure to record the mass of all empty and full volumetric flasks.

Week 2: Quantifying Cu in solutions using the Flame Atomic Absorption

1. Finish any remaining dilutions

You should have the following standards prepared in acid-washed volumetric flasks:

6 standards
15 pennies (10 pennies + 5 duplicate dilutions)
8 individual unknowns (4 unknowns x duplicate dilutions)
26 samples total for Flame AA analysis

2. Running samples

Plan to spend 30 minutes setting up the AA instrument and 30 minutes before your data comes out. Groups will make appointments for the AA based on readiness at the beginning of class.

a. Do QC by running Atomic absorption measurements of samples, individual unknowns, and standards will be made in random order. To check instrument performance, periodically (every 10 samples or so) re-run a blank and a mid-concentration standard.

b. Run a fe samples twice so you can assess precision of AA instrument.

c. Plot your data from the calibration curve from the AA data before leaving class to be sure your calibration curve is linear (R^2>0.98).

Calculations

Reducing your raw data

1. Plot your calibration curve. Be sure to include error bars for the y-axis, determine the line of best-fit, R-squared value.
2. Calculate the concentration of Cu and the error (S_x) for each penny and your unknowns (in ppm)
3. Calculate the original weight % Cu in each penny. Propagate your error (S_x) from your absorbance measurements for each penny.
4. Calculate the initial concentration of Cu in your unknown Cu solution.

QA/QC calculations

Similar to a case 3 t-test, a standard deviation can be estimated from many pairs of duplicate data using the formula below. $\mathbf{d_i}$ values are the differences in the two results for mass of Cu in each penny for each duplicated sample and **k** is the number of duplicate observations.

$$\text{Standard Deviation} = \sqrt{\frac{\sum d_i^2}{2k}}$$

Use the final mass of copper in the penny calculated above (question 3) for the calculations below.

5. Calculate the standard deviation using the formula above for duplicate AA measurements of several pennies.
6. Calculate the standard deviation using the formula above for duplicate dilutions of penny and unknown solutions.
7. Calculate the average and standard deviation of the copper weight percent all 10 pennies.

Hypothesis testing

8. Make a plot of weight % Cu as a function of manufacture year.
9. Perform a t-test to determine if the slope is statistically different from zero at the 95% CI

This t_{calc} is only valid for testing whether the slope of the regression line is zero.

$$t_{calc}\ (\text{Slope}) = m/s_m$$

Both m and s_m can be obtained from the regression output in Excel. Look up t_{tab} in table for the 95% CI and n-2 degrees of freedom. If $t_{calc} > t_{tab}$, reject the null hypothesis (the slope is statistically

different from zero at 95% confidence).

Discussion Questions

1. Do you observe a trend in Cu content as a function of manufacture year? Is this trend statistically significant?
2. What is the largest source of random error in these measurements? You have calculated a standard deviation for duplicate Flame AA data, for duplicate dilutions, and for the mass of all pennies. Which of these three sources contributed the most random error? And what could be done to minimize it?

Lab write-up

1. **Purpose:** write a 1-sentence purpose for the entire lab.
2. **Introduction:** Give a brief introduction to the problem being addressed. Be sure to cite any materials you use.
3. **Results:** Briefly discuss your results in text, shown in figures and tables:
    a. Figure 1: Calibration curve (include error bars, line of best fit, and r-squared values)
    b. Figure 2: Copper weight percent vs manufacturing year (include error bars and results from t-test)
    c. Table 1: Mass pennies as a function of manufacture year (be sure to include errors)

sample	Mass penny (g)	$[Cu^{2+}]$ in solutions (ppm)	Mass Cu in penny (g)	Weight % Cu
1983				
1984				
1992				
Unknown(s)				

    d. Table 2: Standard deviations calculated from duplicate data

Source	St. dev.
Flame AA- instrument error	
Bench chemistry- dilution error	
Variation in mass of pennies	

    e. Table 3: Concentration in unknown

Unknown concentration	St. dev.
measured using Flame AAS	
Provided by TA	
% Recovery	

4. **Discussion:** respond to the questions above in paragraph form. Devote 1-2 paragraphs to discuss each question from week 2 and 1-2 paragraphs for the whole class data.
5. **Conclusion:** Write a short paragraph concluding what you learned.
6. **References**

7. **Group dynamics:** let me know how you feel your group worked together.
8. **Appendix 1- Calculations:** Send a NEAT and ORGANIZED spreadsheet to instructor.