Name:_____

Analysis of Silver in an Alloy

Pre-Lab Questions:

(complete in lab notebook)

1. Define *precipitate, soluble*, and *insoluble*.

2. What is the difference between qualitative and quantitative analytical methods?

3. Why is it possible to analyze the silver content of a silver-copper alloy by precipitating with chloride ion?

4. Is there any other ion besides chloride that could be used in this procedure? If so, why would this ion work?

5. A silver-copper alloy had a mass of 0.1264g. When the alloy was dissolved and the silver precipitated as silver chloride, the precipitate had a mass of 0.1375g. Calculate the percent of silver in the alloy. Show your calculations.

6. If the silver chloride is not dry when its mass is determined, will the calculated percent of silver in the alloy be too high or too low? Explain.

Introduction:

The US Mint has found a stockpile of dimes that are so worn that the dates are unreadable. To determine how old these coins are, and their value, the government has hired you, a chemical engineer, to perform an analysis that will allow you to determine the metallic composition of the dimes. Since the dimes are so worn, it is assumed that they were minted before 1965 when, due to a silver shortage, the mint stopped manufacturing their dimes from a silver-copper alloy and began using copper and nickel.

To perform your analysis, you will first dissolve a portion of the suspect dime in nitric acid, precipitate the silver as silver chloride, and then filter, wash, dry, and mass the silver chloride. Comparing the mass of silver chloride to the mass of the original sample will allow you to calculate the percent of silver in the alloy. This procedure is classified as gravimetric analysis since the results are based on the mass of a product.

Both silver and copper are very non-reactive metals. Neither will dissolve in hydrochloric acid or sulfuric acid. Instead, the "oxidizing agent", nitric acid (HNO_3), is required. In acidic solutions, the nitrate ion (NO_3^-) is an excellent oxidizer and it will oxidize $Ag_{(s)}$ to $Ag^+_{(aq)}$ and $Cu_{(s)}$ to $Cu^{2+}_{(aq)}$.

The reduction product is a colorless gas, nitrogen monoxide (NO), which immediately reacts with oxygen in the air to produce the orange-brown gas nitrogen dioxide (NO_2). The half-reactions are as follows:

$$Ag_{(s)} \rightarrow Ag^+_{(aq)} + e^-$$

$$Cu_{(s)} \rightarrow Cu^{2+}_{(aq)} + 2e^-$$

$$4\ H^+_{(aq)} + NO^{3-}_{(aq)} + 3\ e^- \rightarrow NO_{(g)} + 2\ H_2O_{(l)}$$

Once the silver and copper ions are in solution, they can be separated from each other by precipitating the silver as silver chloride. Silver chloride is very insoluble in water, while copper (II) chloride is soluble. Therefore, addition of chloride ions to the solution will precipitate essentially all of the silver ions and none of the copper ions. The silver chloride precipitate is filtered from the solution.

Chemicals:
silver—copper alloy (pre-1965 dime in 6-8 pieces)
nitric acid, HNO_3, 6 M
baking soda, $NaHCO_{3(s)}$
sodium chloride, $NaCl_{(s)}$

Equipment:
beakers, 100- and 250 mL
Filter flask and Walter's adapter
stirring rod
side-arm Erlenmeyer flask
watch glass
wash bottle
ring stand, ring, wire gauze
crucible tongs
graduated cylinder
Bunsen burner
drying oven
rubber policeman
electronic balance
plastic wrap

Procedure:

Safety Alert

Nitric acid is an extremely corrosive substance that is damaging to skin and eyes. Use great care when you handle it. If you spill any on yourself, wash off with large amounts of water. Neutralize spills with baking soda. Solutions containing silver ions cause dark stains which do not appear for several hours. If you suspect that you spilled any silver ion solutions, immediately clean up with soap and water.

Day 1:

1. Prepare a Filter Crucible

To filter the solution, either a Gooch crucible or a sintered glass filter crucible will be used with a filter flask. Refer to Figure 1 below to see how the crucible, Walter's adapter, and filter flask are assembled.

If you use a Gooch crucible, clean it, place a fiber glass filter pad in the crucible and pull distilled water through the assembly to be sure the filter pad is seated tightly on the bottom of the crucible. If you are using a filter crucible, clean it and rinse it with distilled water using suction. Place the Gooch or sintered glass filter crucible in a clean beaker and dry it in an oven at 110°C. When the crucible is dry, cool it, and determine its mass using a sensitive balance. Be careful to handle the crucible so that no fingerprints will be present.

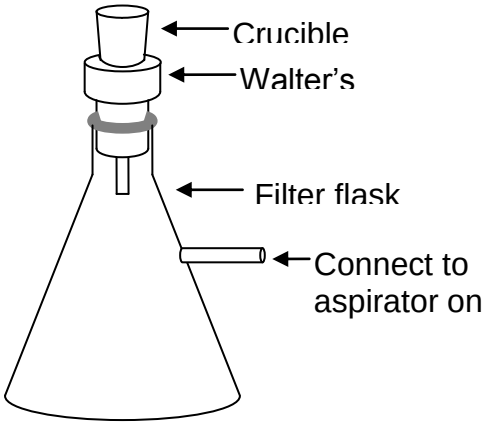

Figure 1. filter flask with crucible and adapter

2. Find the Mass of the Alloy

Obtain a sample of the silver alloy dime that is between 0.1 and 0.5 g. Determine its mass precisely on an analytical balance.

Safety Alert

As the silver-copper alloy dissolves, the gases nitrogen monoxide, NO, and nitrogen dioxide, NO_2, are evolved. Both of these gases are toxic, so this reaction must be carried out in a fume hood.

3. Dissolve the Silver (overnight)

Put the alloy in a clean, labeled, 100-mL beaker. In the fume hood, carefully pour 10 mL of 6M nitric acid over it. Cover the beaker with a watch glass so none of the solution spatters out.

Day 2:

4. Precipitate the Silver

Calculate the amount of sodium chloride that would be necessary to precipitate the silver in your sample, assuming that the sample is 100% silver. Use a balance sensitive to ± 0.01 g to mass out two times this amount of sodium chloride. Dissolve the sodium chloride in 25mL of distilled water. Remove the watch glass from the first beaker, and rinse any moisture on the bottom of the watch glass back into the beaker with your wash bottle to be sure that no silver is lost. To precipitate the silver as silver chloride, slowly add the sodium chloride solution to the dissolved silver. Stir with a stirring rod, and rinse any solution clinging to the rod into the beaker with distilled water. Cover the beaker with Parafilm and allow it to stand overnight. This will allow the precipitate particles to grow larger.

Day 3:

5. Filter Silver Chloride

Attach the filter crucible to a suction flask. Pour some distilled water through the filter with suction to be sure that the filter paper firmly seals the bottom of the funnel. In your wash bottle, add 2 mL of 6 M HNO_3 to 150 mL of distilled water. Label your wash bottle appropriately. Use this as a rinse. The addition of the acid to the rinse water helps to keep the precipitate from “peptizing”, or forming extremely small particles that will run through the filter. Carefully pour the solution above the silver chloride down a stirring rod into the funnel. Wash the precipitate into the funnel with the dilute nitric acid solution. Be sure to get every particle! A rubber policeman on a stirring rod can be used as a squeegee to clean the sides of the beaker. Rinse the precipitate several times with the wash solution.

6. Dry the Silver Chloride and Determine Its Mass

Put the crucible with silver chloride in a clean beaker, cover it with a watch glass, and let it dry in the oven at 110°C for at least 30 minutes. When it is dry and cool, find its mass on the analytical balance. If you have time, dry it an additional 30 minutes and again determine its mass to see if it reached a constant value.

7. Calculate the Percent Silver in the Alloy

From your data, determine the percent of silver in your sample.

Calculations:

Make sure to show at least one example of **each** calculation completed for this lab in your lab notebook.

Analysis:

Answer the following questions in your laboratory notebook.

1. Why is a twofold excess of chloride added to precipitate the silver?

2. Why don't you have to mass the sodium chloride on a sensitive balance?

3. Why is it necessary to wash the precipitate?

4. Will the nitric acid in the wash water interfere with the weight of the silver chloride? (Nitric acid is prepared by dissolving the gas N_2O_5 in water.)

5. If the funnel containing the silver chloride is not cool when its mass is determined, will the calculated percent silver be too high or to low? Why?

6. Why don't we just use hydrochloric acid to both dissolve and precipitate the silver?

7. Why is a Büchner funnel used rather than an ordinary funnel?

8. In September 2011, the price of silver was about $40.00 per ounce. Calculate the price of the silver in the alloy that you analyzed during this lab.