GRAVIMETRIC ANALYSIS OF A CHLORIDE SALT

REFERENCES: *Nelson, J., Chemistry: The Central Science, 3rd edition, Prentice-Hall, 1985*

Typical techniques used in gravimetric analyses by quantitatively determining the amount of chloride in an unknown sample will be illustrated.

Apparatus and Chemicals required:

250 mL beakers (3)	filter paper	1.5 g unknown chloride
100 mL or larger beakers (3)	weighing paper	funnels (3)
plastic wash bottle	0.125 M $AgNO_3$	stirring rods (3)
	6 M HNO_3	rubber policemen (3)
	acetone, 60 mL	funnel support
	Bunsen burners (3)	watch glasses (3)
	ring stands and rings	sample bottle
		wire gauze (3)

DISCUSSION

Quantitative analysis is that aspect of analytical chemistry which is concerned with determining how much of one or more constituents is present in a particular sample of material. We have already seen how information such as percentage composition is essential to establishing formulas for compounds. Two common methods used in analytical chemistry are gravimetric and volumetric analysis. Gravimetric analysis derives its name from the fact that the constituent being determined can be isolated in some weighable form. Volumetric analysis, on the other hand, derives its name from the fact that the method used to determine the amount of a constituent involves measuring the volume of a reagent. Usually, gravimetric analyses involve the following steps:

1. Drying and then accurately massing[1] representative samples of the material to be analyzed.
2. Dissolving the sample.
3. Precipitating the constituent in the form of a substance of known composition by adding a suitable reagent.
4. Isolating the precipitate by filtration.
5. Washing the precipitate to free it of contaminants.
6. Drying the precipitate to a constant mass (to obtain an analytically weighable form of known composition).
7. Calculating the percentage of the desired constituent from the masses of the sample and precipitate.

[1] The terms “massing” and “weighing”, just as the terms “mass” and “weight”, are used interchangeably in chemistry, despite the fact that they are NOT the same (strictly speaking).

Although the techniques of gravimetric analysis are applicable to a large variety of substances, we have chosen to illustrate them with an analysis that incorporates a number of other techniques as well. Chloride ion may be quantitatively precipitated from solution by the addition of silver ion according to the following ionic equation:

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$$

Silver chloride is quite insoluble (only about 0.0001 g of AgCl dissolved in 100 mL of H_2O at 20°C); therefore, the addition of a silver nitrate solution to an aqueous solution containing chloride ion precipitates AgCl quantitatively. The precipitate can be collected on a filter paper, dried, and weighed. From the mass of the AgCl obtained, the amount of chloride in the original sample can then be calculated.

This experiment also illustrates the concept of stoichiometry. As we know, stoichiometry is the determination of the proportions in which chemical elements combine and the mass relations in any chemical reaction. In this experiment stoichiometry means specifically the mole ratio of the substances entering into and resulting from the combination of Ag^+ and Cl^-. In the reaction of Ag^+ and Cl^- in the above equation, it can be seen that 1 mole of chloride ions reacts with 1 mole of silver ions to produce 1 mole of silver chloride. Thus

$$\text{Moles } Cl^- = \text{moles AgCl} = \frac{gramsAgCl}{MM_{AgCl}}$$

$$\text{Moles } Cl^- \times MM_{Cl} = \text{grams } Cl_{\text{sample}}$$

$$\%\text{Cl in sample} = \frac{gramsCl_{sample}}{mass_{sample}} \times 100$$

where MM represents molar mass

PROCEDURE

Mass by subtraction[2] (to the nearest 0.0001 g) about 0.1 to 0.2 g of your unknown using a sample bottle equipped with a ground-glass fitting; place the unknown directly into a clean 250 mL beaker[3]. Label the beaker "1" with a pencil and record the sample mass AND the beaker number on your report sheet. Add between 75 and 100 mL of distilled water and 1 mL of 6 M HNO_3 to the beaker. Repeat with sample numbers 2 and 3 and label the Beakers "2" and "3". Stir each of the solutions with three <u>*different*</u> glass stirring rods until all of the sample has dissolved. <u>**Leave the stirring rods in the beakers.**</u>

While stirring one of the solutions add to it about 30 mL of 0.125 M $AgNO_3$ solution. Place a watch glass over the beaker. Warm the solution gently with your Bunsen burner

---

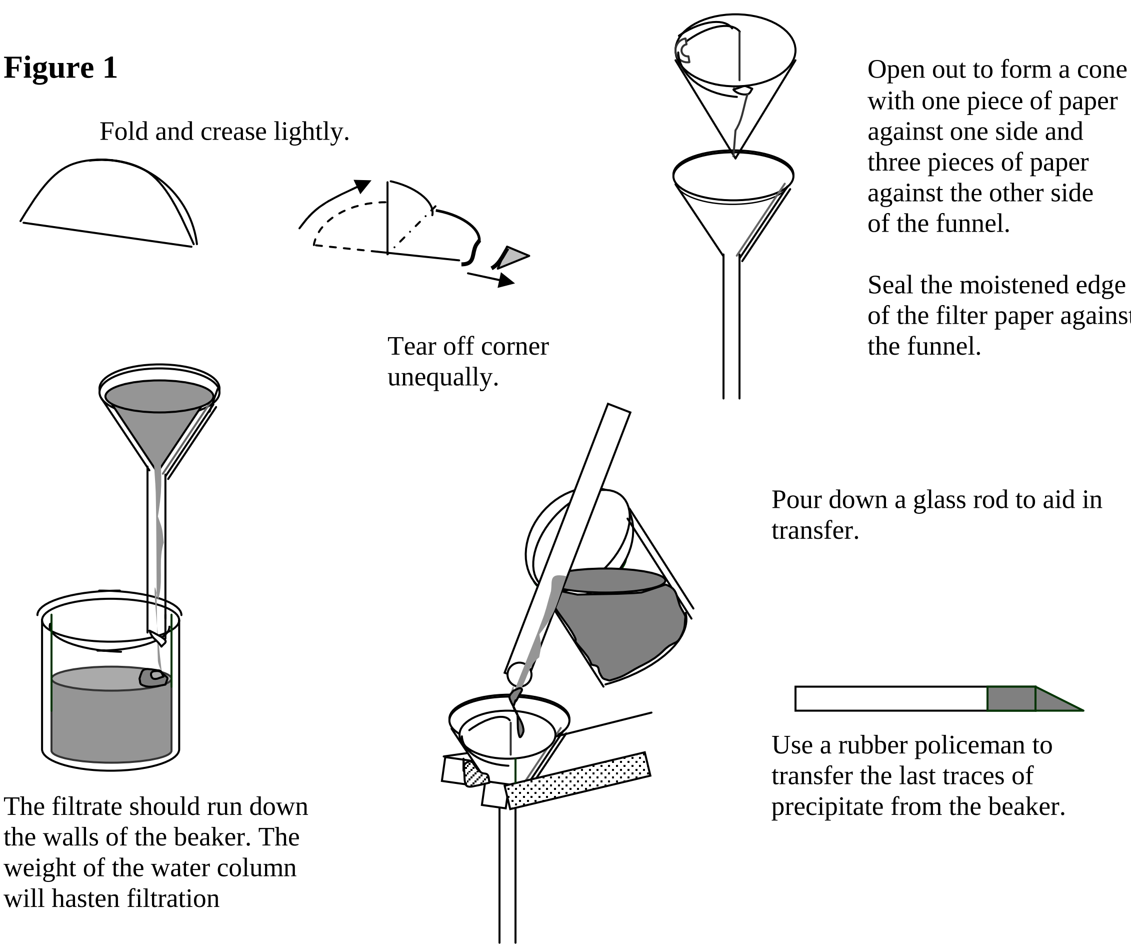
[2] This procedure is accomplished as follows: zero out your semi-micro analytical balance. Place your sample bottle (also called a *weighing bottle*) on the balance and record the weight. Remove the weighing bottle and immediately place some of the sample (between 0.1 and 0.2 g) into your first beaker. Re-weigh your sample bottle and record the new weight. Repeat this procedure two more times.

[3] This is a one-way operation! Do NOT remove sample from the beaker once it has been removed from the weighing bottle!

and keep it warm for approximately 30 minutes (this can be done using either a Bunsen burner or a hot place). **Do not boil the solution!**

Obtain a filter paper (three of these will be needed) and mass it accurately ($\pm 0.0001 g$) after folding and tearing off one specific corner as illustrated in Figure 1 and fit it into a glass funnel. Be certain that you open the filter paper in the funnel so that one side has three pieces and one side has one piece of paper against the funnel—not two pieces on each side. ***Be certain that you weigh the paper after it has been folded and torn, not before.*** Wet the paper with distilled water to hold it in place in the funnel. Completely and quantitatively transfer the precipitate and all the warm solution form the beaker onto the filter using a rubber policeman and a wash bottle to wash out the last traces of precipitate. The level of solution in the filter funnel should always be below the top edge of the filter paper. Wash the precipitate on the filter paper with two or three 5 mL portions of water from the wash bottle.[4] Finally, pour three 5 mL portions of acetone through the filter (methanol can also be used; you will be instructed as to which you will use). **KEEP THE ACETONE AWAY FROM OPEN FLAMES BECAUSE IT IS HIGHLY FLAMMABLE!** Remove the filter paper; place it on *numbered* watch glass; and store it in your locker until the next period.

Figure 1



[4] Check for completeness of precipitation by adding a few drops of $AgNO_3$ solution to the clear filtrate. If it clouds you must re-filter. Talk to your instructor before going on!

Repeat the above processes with your other two samples, being sure that you have numbered your watch glasses so that you can identify the samples. The precipitated AgCl must be kept out of bright light because it is photosensitive and slowly decomposes in the presence of light as follows:

$$2\ AgCl\ (s) \xrightarrow{h\nu} 2\ Ag(s) + Cl_2(g)$$

In the next period, when the AgCl is thoroughly dry, weigh the filter papers plus AgCl and calculate the mass of AgCl. From these data calculate the percentage of chloride in your original sample.

Name___________________________________

GRAVIMETRIC CHLORIDE REPORT SHEET

Unknown Number____________

	Trial 1	Trial 2	Trial 3
Mass of sample bottle and contents **before** removal of sample			
Mass of sample bottle and contents **after** removal of sample			
Mass of sample			
Mass of filter paper plus AgCl precipitate			
Mass of filter paper			
Mass of AgCl			
Mass of Cl in original sample (show calculation below)			
Percent chloride in original sample (show calculation below)			
Average percent chloride (show calculation below)			
Standard deviation (show calculation below)			

Show calculations.

Mass of chloride in original sample from mass of AgCl for one trial.
Percent chloride in original sample for one trial.
Calculate average for all trials.
Calculate standard deviation for all trials.

Name____________________________________

QUESTIONS

1. Barium can be analyzed by precipitating it as $BaSO_4$ and weighing the precipitate. When a 0.2000 g sample of a barium compound was treated with excess H_2SO_4, 0.121 g of $BaSO_4$ formed. What is the percentage of barium in the compound?

2. An impure sample of table salt that weighed 0.6543 g, when dissolved in water and treated with excess $AgNO_3$ form 1.258 g AgCl. What is the percentage of NaCl in the impure sample? Assume all the chloride came from NaCl.

3. What effect (if any) would the following have on your results?
   a) You did not completely dry your AgCl.

   b) The **actual** molarity of $AgNO_3$ solution was different than that reported on the bottle.

   c) Your NaCl sample was contaminated with 1% lead(II) nitrate.