GRAVIMETRIC ANALYSIS OF A CHLORIDE SALT

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)
- 0.125 M AgNO₃
- 3 beakers—any 100 mL or larger
- 0.125 M HNO₃
- plastic wash bottle
- acetone, 60 mL
- stirring rods (3)
- distilled water
- watch glasses (3)
- From Stockroom:
- funnels (3)
  - sample bottle
  - rubber policeman (3)
- filter paper
- funnel support
- weighing paper
- 1.5 g unknown chloride sample
- ring stand, rings
- wire gauze
- Bunsen burner
- balance

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 weighing 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 to a constant mass (to obtain an analytically weighable form for known composition.)
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.
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:

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

Silver chloride is quite insoluble (only about 0.0001 g or AgCl dissolves in 100 mL or H$_2$O at 20° C); therefore, the addition of silver nitrate solution to an aqueous solution containing chloride ion precipitates AgCl, quantitatively. The precipitate can be collected on a filter paper, dried, and massed. 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 from stoichiometry,

$$\text{Moles Cl}^- = \text{mole AgCl} = \frac{\text{grams AgCl}}{\text{molar mass AgCl}} \quad \text{------------ (1)}$$

$$\text{mass of Cl in sample} = (\text{mole Cl}^-) \times (\text{molar mass of Cl}) \quad \text{-------- (2)}$$

$$\%\text{Cl in sample} = \frac{(\text{mass of Cl in sample}) \times 100}{\text{mass of sample in gram}} \quad \text{---------- (3)}$$

**PROCEDURE**

**Mass by subtraction:**

i) Using a weighing paper weigh about 0.1 to 0.2 g of your unknown sample on a top loading balance. (remember to tare the paper mass)

ii) Transfer the sample from the paper into a weighing bottle and weigh it accurately.

iii) After transferring the sample from the weighing bottle carefully into a clean 250 mL beaker, weigh the empty bottle alone accurately.

**Precipitation:**

Label the beaker #1 with a pencil. 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 different glass stirring rods until all of the sample has dissolved. Leave the stirring rods in the beakers. While stirring one of the solutions, add to it about 30 mL of 0.125 M AgNO$_3$ solution slowly in drops. Place a watch glass over the beaker. Cover the beakers and watch glasses with foil to protect from light.

Warm the solutions gently with your hot plate and keep it warm for approximately 30 minutes.

**Do not boil the solution!**
Filtration:
A. Obtain a filter paper (three of these will be needed).
B. Fold the paper as illustrated in Figure 1.
C. Be certain you mass the paper accurately after it has been folded and torn, not before.
D. 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.
E. 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 from 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. (Check for completeness of precipitation by adding a few drops of the AgNO₃ solution to the clear filtrate. If it clouds you must re-filter.)

Finally, pour three 5 mL portions of acetone through the filter. KEEP THE ACETONE AWAY FROM OPEN FLAMES BECAUSE IT IS HIGHLY FLAMMABLE! Remove the filter paper; place it on a numbered watch glass; and store it in your locker until the next period.

Repeat the above processes with your other two samples, being sure that you have numbered your watch classes 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:

\[ 2\text{AgCl}_{(s)} \rightarrow 2\text{Ag}_{(s)} + \text{Cl}_2(g) \]

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

Unknown number __________

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<tr>
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<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 3</th>
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<tr>
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<td>Mass of weighing bottle after transfer of sample</td>
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<td>Mass of sample</td>
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<td>Mass of filter paper + AgCl</td>
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<td>Mass of AgCl</td>
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<td>Moles of Cl⁻ (use eqn 1)</td>
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<td>Mass of Cl⁻ (use eqn 2)</td>
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Work:
REFERENCES:


QUESTIONS:

1. Barium can be analyzed for by precipitating it as BaSO₄ and mass the precipitate. When a 0.2000 g sample of a barium compound was treated with excess H₂SO₄, 0.121 g of BaSO₄ formed. What is the percentage of barium in the compound?

2. An impure sample of table salt that massed 0.6543 g, when dissolved in water and treated with excess AgNO₃, formed 1.258 g of AgCl. What is the percentage of NaCl in the impure sample?