Identifying an unknown chloride salt by gravimetric analysis

Summary

Gravimetric analysis will be performed to identify an unknown chloride salt. This method of analysis allows for a quantitative determination of the mass percent of chlorine in the unknown through precipitation of the chloride ions in the form of silver chloride. The mass percent can then be calculated and compared to the theoretical mass percent of chlorine in the candidate unknown salts.

Introduction

Quantitative analysis is that aspect of analytical chemistry concerned with determining how much of one or more constituents are present in a particular sample of material. Information such as percentage composition is essential in analytical chemistry for determining chemical formulas for compounds. Two common methods for quantitative analysis are gravimetric analysis and volumetric analysis.

Gravimetric analysis derives its name from the process of isolating the desired constituent in weighable form. Volumetric analysis derives its name from the process of measuring the volume of a reagent. Gravimetric analysis, in short, involves changing one compound containing the constituent into another compound containing that constituent. From the mass of the new compound, the mass percent of the constituent of interest in the unknown sample can be determined,

In this experiment, silver chloride (MW = 143.37 g/mole) will be produced from an unknown chloride compound. The percent chloride will then be determined based on the amount of silver chloride recovered from the precipitation reaction. Vacuum filtration will be used in order to recover the precipitate.

The unknowns for this experiment consist of the following three chloride salts:

Salt Name	Formula	Formula Weight (g/mole)
Sodium Chloride	NaCl	58.49
Potassium Chloride	KCl	74.60
Magnesium Chloride Hexahydrate	$MgCl_2 \cdot 6H_2O$	203.30

Experimental

1.   Obtain a vial containing an unknown chloride salt.   Be sure to note the identification letter or number of your unknown.

2.   Weigh out about 0.100 ± 0.005 g of your unknown into a clean weigh boat.

3.   Quantitatively transfer the solid into your Erlenmeyer flask by pouring the solid into the flask and then washing the weigh boat into the flask several times with distilled water.

4.   Add about 25 mL of distilled water to dissolve the remaining solid in your flask. If the solid does not completely dissolve, add a few additional milliliters of distilled water.

5.   Next, precipitate the chloride as silver chloride by adding an excess of silver nitrate. Add about 20 mL of 0.15 M silver nitrate ($AgNO_3$) to your flask and swirl to mix thoroughly. Then let the precipitate settle to the bottom of your flask.

6.   Prepare to filter your precipitate by locating your fritted (Gooch) crucible and obtaining a crucible holder, filter paper/pad and filtering flask. **Place the filter in the crucible and record the mass.**

7.   Set-up the filtration apparatus as demonstrated by your laboratory instructor. Apply suction, and slowly pour approximately 10 mL of distilled water through the crucible. Use a stirring rod to help seat the filter in the crucible. Be sure that the filter covers all of the holes and that it is properly seated onto the crucible so that no solid can pass around the outside edges of the filter.

8.   While the suction remains on, carefully filter the precipitate by pouring it slowly from the Erlenmeyer flask into the crucible. Use small portions of distilled water to wash your Erlenmeyer flask to ensure your have transferred all of the solid precipitate to the crucible. Wash the precipitate in the crucible, by slowly adding two 10 mL portions of the distilled water.

9.   Break the vacuum at the flask by disconnecting the tubing from the flask while the aspirator is still running. Transfer the filtrate (liquid) into a clean beaker and test for complete precipitation by adding a few drops of the silver nitrate solution. If all of your chloride has precipitated, you should see no changes after adding more silver nitrate. If you observe additional precipitation of silver chloride, see you instructor for assistance.

10.   Reconnect the vacuum and turn on the suction. Wash the precipitate by slowing adding three 10 mL of acetone to the crucible. Be sure to allow the entire volume to filter through the crucible before adding the next portion of acetone.

11.   While the suction remains on, **WAIT 15 MINUTES** to allow the precipitate to dry. You must wait 15 minutes in order to obtain meaningful results for this experiment. You will not be able to indentify your unknown if your precipitate is not dry.

12.   Break the vacuum at the flask by disconnecting the tubing from the flask while the aspirator is still running. Remove your crucible and determine the mass of your crucible, filter, and solid precipitate.

13.   After recording the mass of your solid product, transfer the solid and filter paper to the solid water container located in the fume hood. Your liquid waste should go in the proper waster container located in the hood. **The waste from this experiment DOES NOT GO DOWN THE DRAIN.**

14.   Enter the following data into the lab computer: ID of unknown, mass of unknown used, mass of solid AgCl precipitate recovered

Calculations

First you will need to calculate the percent chlorine in your unknown sample. Determine the mass of your silver chloride precipitate by difference. Next, you will need to use stoichiometry to calculate the mass of chlorine in your solid AgCl precipitate. Finally, since you know the mass of the unknown salt you began with you can determine the mass percent of chlorine in your unknown:

$$mass\ percent\ chlorine(\%) = \frac{mass\ of\ chlorine\ in\ unknown\ (g)}{mass\ of\ unknown\ (g)} \times 100$$

In order to indentify the unknown you will need to compare the experimental mass percent of chlorine with the theoretical mass percent of the three candidate salts. For example, if LiCl was a candidate unknown (it is not for this experiment) you know that for every mole of LiCl there is 1 mole of Cl present. The formula weight of LiCl is 42.44 g/mole, so one mole of LiCl weighs 42.44 g.   In that one mole of LiCl, there is one mole of Cl weighing 35.5 g. Therefore, the mass percent of chlorine in LiCl is:

$$mass\ percent\ chlorine\ in\ LiCl\ (\%) = \frac{35.5\ g}{42.44\ g} \times 100 = 83.6\%$$