Gravimetric Analysis of Chloride

I. Introduction

The amount of chloride ion in a substance can be determined by precipitating the soluble chloride ion present with silver ion. The amount of chloride ion present will be related to the mass of silver chloride formed.

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\text{Ag}^+_{(aq)} + \text{Cl}^-_{(aq)} \rightarrow \text{AgCl}_{(s)}
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II. Procedure

A. Preparation of Filter Crucibles (Sintered-Glass)

1. Use four filter crucibles of medium (M) porosity. Do not use crucibles marked C for coarse porosity.

2. Previously, the crucibles have been used to filter silver chloride (purple or gray, ammonia soluble). First, remove any bulk precipitate with a rubber policeman and dispose of it in a "Silver Collection" jar. Follow this with chemical cleaning. To remove silver chloride, invert the crucibles in a large beaker in a fume hood and add one or two mL of concentrated ammonia to each. Allow to soak until clean. Carefully remove the crucibles from the beaker and soak in water before removing from the fume hood. Set up a suction filtration apparatus and wash the crucible several times with water and finally with deionized water. Empty the suction flask before proceeding. Stains that are not removed by this cleaning process should not interfere with your measurements. Number, in pencil, each crucible on the frosted glass area.

3. Dry the crucibles in an oven at 120°C for about 1 hour, using a beaker, glass hooks, and watch glass (see figure). A ribbed watch glass can be used in place of the glass hooks.

4. Remove the dried crucibles from the oven and let them cool for about one minute on the counter top. With clean crucible tongs transfer the crucibles to the desiccator. After allowing the crucibles to cool to room temperature for 30 minutes, weigh accurately. Repeat the drying operation for 1/2 hour and again determine the crucible mass. Continue this drying and weighing procedure until each crucible mass is constant within ± 0.5 mg. Since the mass of AgCl precipitated will be approximately 600 mg, this represents a relative error of less than 1 ppth. When the masses obtained for a given crucible no longer show any particular trend (the values may fluctuate up and down), take the average of these values to be the 'true' mass.
B. Precipitation Procedure

1. Dry the unknown chloride in a weighing bottle (with top off in a covered beaker as was done for the crucibles) at 120°C for 2 hours while preparing the filter crucibles. Store the dried unknown in a desiccator with the weighing bottle top in place. Weigh accurately 4 samples of about 0.25 g (to four significant figures) each into clean 400-mL beakers numbered the same as the crucibles. (The samples only need to be 0.25xx g ± 15%).

2. Add 150 mL of chloride-free deionized water and acidify with 1 mL of 6 M HNO₃. Stir. Use a separate stirring rod for each beaker, and leave it in the beaker throughout the procedure.

3. Assume the sample is pure sodium chloride and calculate a theoretical maximum volume of 0.2 M silver nitrate needed to precipitate the chloride ion. Prepare the required volume (plus about 10% excess) of 0.2 M AgNO₃ from 1.0 M AgNO₃ by dilution. Store the 0.2 M AgNO₃ solution in your brown reagent bottle.

4. Add slowly, with stirring, the theoretical maximum volume (including the 10% excess) of 0.2 M silver nitrate to each sample. Adjust this volume accordingly if the sample mass is significantly different than 0.25 g. Note the volume of 0.2 M AgNO₃ added. Store your samples covered in your locker until the next period.

5. Once the precipitate has coagulated on the bottom of the beaker so that the upper portion of the solution is clear, test for complete precipitation of chloride by adding a few drops of 0.2 M silver nitrate and observing if additional precipitate appears. If it does, add more 0.2 M silver nitrate solution (5% of the initial amount), stir thoroughly and store covered in your locker until next period. Small particles will form on the beaker walls and some will float on the surface. After sufficient coagulation, test again for complete precipitation.

C. Filtration and Weighing

1. Prepare a wash solution by adding 1.5 mL of 6 M nitric acid to 200 mL of deionized water. You may want to put this solution into your wash bottle for convenience.

2. Place a weighed filter crucible in the suction filtration apparatus and apply gentle suction. Decant the supernatant solution from the corresponding beaker through the crucible, retaining the precipitate in the beaker. Add about 10 mL of the wash solution to the beaker with the retained precipitate and agitate to wash the precipitate. Let it settle, and decant again through the crucible. Repeat twice.
3. Now bring the precipitate onto the filter, using small portions of the wash solution for transfer. Remove any solid particles adhering to the beaker with a rubber policeman. Wash the precipitate in the crucible with several 5-mL portions of wash solution. Collect about 1 mL of the last washing in a test tube and **test it for silver ion by adding a drop of 6 M HCl**. Wash again, if a precipitate is noted. When the filtrate in the filter flask tests negative for silver ion, it indicates that all excess AgNO₃ adsorbed on the surface of the precipitate has been removed by the washing and replaced by HNO₃. The adsorbed HNO₃ causes no error since it will be volatilized during the drying of the precipitate.

4. Completely drain each crucible with strong suction. Then place the crucibles with precipitate into an oven and dry to constant mass as in the earlier procedure (II.A.3-4).

5. At the conclusion of the experiment, **place the precipitate in a "Silver Collection" jar** and clean your crucibles by dissolving any remaining AgCl precipitate in 5-10 mL of concentrated NH₃(aq) (do this in a fume hood).

### III. Calculations

Compute the gravimetric factor for converting mass of precipitate to mass of chloride. From the masses of the crucibles with and without precipitate, calculate the masses of silver chloride obtained. Using the gravimetric factor, calculate the mass of chloride in each sample of unknown and the corresponding mass percent chloride. If one of the results appears questionable, test it for rejection. The precision of your results should be 1-3 ppth.

### IV. Results

Report each result, the mean and RMD of the mass percent chloride in your sample, using the appropriate unknown report form, to the instructor.