Experiment I.2: Determination of Chloride by the Mohr Method

This is two-lab periods experiment. During the first class you standardize a silver nitrate solution. During the second lab you analyze your unknown. You **have to leave** your **unknown** in an oven to be dried during the first class (in a weighting bottle; mark this bottle with your initials).

ALL of the data have to be recorded in your notebook

A silver nitrate solution is standardized using a primary-standard sodium chloride. An unknown sample is then analyzed for chloride, and its concentration is determined using this standardized silver nitrate solution.

The Mohr method uses CrO_4^{2-} as an indicator. A precipitate of Ag_2CrO_4 forms in the presence of a slight excess of Ag^+ and signals the end point of titration. The color changes from a yellow to a brownish-yellow. The change can be detected most precisely when the color in the titration flask is compared to a reference color. Therefore, a mixture containing CrO_4^{2-} indicator in a suspension of $CaCO_3$ (simulating a precipitate of AgCl) is used. An indicator blank is prepared using a portion of this mixture. The blank provides a correction for the slight difference between the end point and the equivalence point, a systematic error (this difference is **not** always observed).

Preparation

Read section on volume measurements in your text, especially the material on measurement of an aliquot and directions for use of a burette. Read very carefully the chapter on precipitation titrimetry in your textbook.

Use **ONLY deionized** water in this experiment!

Preparation

- Oven-dried NaCl (in a desiccator) and your unknown are provided. Remember to leave your unknown in an oven at the end of the first class (it will be dried for you and ready for your next class).
- Titrant: Prepare 500 mL of approximately 0.1 M $AgNO_3$ by diluting 100 mL of the 0.5 M stock solution (100 mL of the stock solution and 400 mL of deionized water) – use the cylinder for these measurements. Store this light-sensitive solution in a dark bottle and keep it in your locker away from any light when it is not in use. Use 1 L dark bottle. This solution is your titrant; this is the only solution that you put into the burette.
- Reference and Blank: Add 4 mL of an indicator (0.1 M K_2CrO_4 – it is stored under the hood) and approximately 0.5 g of $CaCO_3$ to 200 mL of water (cylinder). Use half of this mixture for the color reference (before titration) and the other half to determine the indicator blank (see later comment). Use two beakers for these two solutions.

Procedure for Standardization of Silver Nitrate Solution

- Standard NaCl Solution: Transfer a 1.0 g (analytical balance, record the mass to the nearest 0.1 mg) sample of standard sodium chloride into a small funnel placed in the neck of a 100 mL volumetric flask (this flask does not have to be dry!). Make sure that the funnel is dry, and that the salt goes into the flask without accumulating in the funnel. Carefully rinse any crystals of the sample into the flask. Use only deionized water here. The **sample** should be

completely dissolved BEFORE filling the flask up to the mark. Volumetric flasks are not well design for mixing. After filling to the mark, close with the stopper, invert the flask repeatedly and let the air bubbles do the mixing. Reagents which are NOT thoroughly mixed together will form **inhomogeneous** solutions and lead to **serious errors**. You will use four 25 mL aliquots of this standard solution (see below). Since 1.0 g of NaCl is about 16 mmoles, each 25 mL aliquot will contain exactly 1/4 of the total, *i.e.*, 4 mmoles of Cl^-. This will require approximately 40 mL of the 0.1 M silver nitrate solution from a burette.

- Preparation for Titration: Rinse the burette with a small portion of $AgNO_3$ solution (approx. 0.1 M solution that you have prepared) before filling it up. This will remove the water that might contribute to an error. Fill the burette up the 0.00 mL mark with the $AgNO_3$ solution. Titrate the half of the blank solution first (takes one or two drops). It will help you to visualize the end point. Leave this blank sample on a side for the titration reference. You are ready for titration.
- Aliquots: Rinse your pipette twice with very small portions of the standard NaCl solution. Pipette a 25 mL aliquot into a 250 mL Erlenmeyer flask, add 25 mL deionized water (graduated cylinder), 2 mL of indicator (0.1 M K_2CrO_4 solution; **DO NOT** forget this indicator). Prepare two more aliquots the same way.
- Titration: Fill the burette to the 0.00 mL mark. Record the starting volume. Titrate each aliquot with the silver nitrate solution. Be very careful with the first aliquot. Start with the additions of approximately 1 mL (this does not have to be exact) of the $AgNO_3$ solution and mix the solution vigorously. When you observe first color changes, start adding the solution drop–by–drop. **Color change** that does not disappear for a few minutes indicates the end point of titration (compare the color with two blanks, first before titration, yellow, and the second one after titration; brownish–yellow).
  The other aliquots should be titrated relatively fast (with mixing) close to the end point (approx. 5 mL, and then drop–by–drop.
  Record all the volumes (initial, finals) to the nearest 0.02 mL.

Determination of % Chloride in the Unknown

Weigh out a charge of approximately 150 mg of the unknown (analytical balance, ±0.1 mg) and transfer it directly into 250 mL Erlenmeyer flask. Dissolve it in approximately 50 mL of water, add 2 mL of indicator and titrate. Record the volume of $AgNO_3$ solution (initial and final).

The final volume of the $AgNO_3$ solution should be close to 30 mL. Adjust the size of your unknown (more or less than 150 mg) and repeat the process with your unknown two more times.

Housekeeping

Dispose of AgCl from titrations and excess $AgNO_3$ into provided containers. Return stock solution bottles.

Report

Report the average percent chloride in your unknown, the percent chloride found with each determination, the standard deviation, and the relative standard deviation (this report page should also include your name, the date of the experiment, the date of the report, and the title of the experiment). Raw data and calculations MUST be in your notebook for inspection upon request.