Experiment 24

Determination of Chloride Ion in Sea Water Via Selective Precipitation

Materials:

5 mL pipet, 250 mL Erlenmeyer flask, 50 mL buret, 0.1 M K_2CrO_4 solution, standard $AgNO_3$ solution, 2 weighing bottles, weighing boat, 50 mL graduated cylinder, 10 mL graduated cylinder, 2 50 mL beakers.

Discussion:

In this experiment you will make use of the principle of selective precipitation, a process which takes advantage of differing solubilities to selectively remove one of several ions from solution. You will now be able to apply some of the solubility product calculations you have been concerned with in the classroom. To show how this concept applies to this particular analysis, consider a solution 0.1 M in chloride and 0.01 M in chromate. By the slow addition of aqueous silver nitrate, the chloride can be precipitated as silver chloride leaving the chromate in solution. Consider the following:

$$AgCl\ (s) \rightleftarrows Ag^+\ (aq) + Cl^-\ (aq) \qquad K_{sp} = 1.7 \times 10^{-10} = [Ag^+][Cl^-]$$

$$Ag_2CrO_4\ (s) \rightleftarrows 2\ Ag^+\ (aq) + CrO_4^{2-}\ (aq) \qquad K_{sp} = 2.0 \times 10^{-12} = [Ag^+]^2[CrO_4^{2-}]$$

Using these K_{sp} values, the molar solubilities can be calculated as1.3×10^{-5} for silver chloride and 7.9×10^{-5} for silver chromate. Thus, even though both silver chloride and silver chromate are "insoluble" compounds, silver chloride is the less soluble of the two.

From the K_{sp} values it can be shown that in order to precipitate silver chromate from the 0.01M chromate solution, the $[Ag^+]$ in solution must be greater than 1.4×10^{-5} M, i.e.

$$\left[Ag^+\right]^2 = \frac{K_{sp}}{\left[CrO_4^{2-}\right]} = \frac{2.0\text{x}10^{-12}}{.01}$$

and, therefore,

$$[Ag^+] = 1.4 \times 10^{-5}\ M$$

By the time this silver ion concentration has been reached, all the chloride ions will have precipitated as silver chloride. This can be shown by:

$$K_{sp} = 1.7 \times 10^{-10} = [Ag^+][Cl^-] = [1.4 \times 10^{-5}][Cl^-]$$

$$\therefore [Cl^-] = 1.2 \times 10^{-5}\ M$$

Therefore, in order to reach a $[Ag^+]$ of this magnitude the $[Cl^-]$ must be less than 1.2×10^{-5} M which means that when determining the percent of the chloride precipitated from solution:

All of the original chloride has been removed when red silver chromate begins to precipitate. Thus our reaction is quantitative and the colored silver chromate serves as a suitable indicator for the equivalence point of the titration.

Procedure: (work individually)

Part A:

Weigh a clean, dry weighing bottle on the analytical balance and pipet 5.00 mL of the unknown seawater sample into this bottle. Weigh the bottle containing the 5.00 mL sample. Record both masses in the Data Section.

Carefully make a quantitative transfer of the weighed seawater sample into a clean 250 mL Erlenmeyer flask. Use small squirts from a distilled water wash bottle to flush out the weighing bottle. Add 50.0 mL of distilled water and 2 mL of 0.1 M potassium chromate solution, which will serve as an indicator, to the Erlenmeyer flask. Titrate the seawater mixture with standard silver nitrate solution.

The silver chloride precipitate formed is white, but appears lemon yellow in the yellow potassium chromate solution. When the precipitation of silver chloride is essentially complete, further addition of silver nitrate solution results in precipitation of red silver chromate. Thus the first appearance of silver chromate is taken as the end-point of the precipitation of chloride. This slight red color changes the bright lemon yellow to a rather muddy (almost mustard) yellow color. This change should be approached carefully and attained with a one or two drop addition of silver nitrate. Record the concentration and volume of silver nitrate solution required.

Repeat the above procedure (Trial 2). Note that the Erlenmeyer flask contents (liquid and precipitate) should be properly disposed of in the waste crock in the hood, NOT down the sink. The white film which remains in the flask will not affect later results, but can be removed using an abrasive cleanser (Comet) or chemicals such as thiosulfate or ammonia solutions.

Part B:

Use a weighing boat to weigh, on the analytical balance, a sample of an unknown solid chloride weighing approximately 0.08 grams. Transfer this solid to a 250 mL Erlenmeyer flask and weigh the "empty" weighing boat. Now proceed as in Part A, adding 50.0 mL of distilled water, 2 mL of 0.1 M potassium chromate solution and titrating with standard silver nitrate solution to a good silver chromate end-point. Your instructor may suggest a duplicate run if time permits.

Name _____ Section _____

Partner _____ Date _____

DATA SECTION
Experiment 24

Part A:

Analytical balance number: _____

	Trial 1	Trial 2
Mass of weighing bottle and seawater	_____	_____
Mass of weighing bottle	_____	_____
Mass of seawater	_____	_____
Concentration of std. $AgNO_3$ solution	_____	_____
Final Buret Reading	_____	_____
Initial Buret Reading	_____	_____
Volume of std. $AgNO_3$ solution used	_____	_____
Number of moles of $AgNO_3$ used	_____	_____
Number of moles Cl^- in 5.00 mL of seawater	_____	_____
Weight % of Cl^- in seawater	_____ %	_____ %

Part B:

Analytical balance number: _____

Unknown number: _____

	Trial 1	Trial 2
Mass of solid chloride and paper	_____	_____
Mass of weighing paper	_____	_____
Mass of solid chloride sample	_____	_____
Concentration of std. $AgNO_3$ solution	_____	_____
Volume of std. $AgNO_3$ solution used	_____	_____
Number of moles of $AgNO_3$ used	_____	_____
Number of moles of Cl^- in solid sample	_____	_____
Weight % of Cl^- in solid sample	_____	_____