

Experiment 16

THE SOLUBILITY PRODUCT CONSTANT OF SILVER ACETATE

Objective

The purpose of this experiment is to determine the solubility product constant of silver acetate (CH_3COOAg) at different temperature.

Lab techniques

- Operating graduated pipet, stirrer/hot plate, gravity filtration, and titration.

Introduction

When excess slightly soluble salt A_mB_n is added to water, an equilibrium is established between the solid and its dissociated ions in solution (16-1). Such a solution is said to be saturated. The solubility product constant, K_{sp} , of this reaction is given by equation 16-2. The K_{sp} value depends on the temperature. At a given temperature, K_{sp} is a constant for a given salt.



$$K_{sp} = [A^{n+}]^m [B^{m-}]^n \quad (16-2)$$

When two ionic solutions with large enough concentrations that the ion product Q ($Q = [A^{n+}]_o^m [B^{m-}]_o^n$) exceeds K_{sp} are mixed, the cations and anions will combine and precipitate. Occasionally, the salt does not precipitate even if the ion product exceeds K_{sp} . In this case, the solution is said to be supersaturated. In a supersaturated solution, precipitation can be induced by agitating the solution or scraping the walls of its container with a glass rod.

There are many ways to determine K_{sp} , including potentiometry, conductivity measurement, or quantitative spectral analysis. In this experiment, we will determine the K_{sp} value of silver acetate (CH_3COOAg , abbreviated as AgOAc) by titration. The solubility equilibrium and solubility product constant expression of silver acetate are given by equations 16-3 and 16-4.



$$K_{sp} = [\text{Ag}^+][\text{OAc}^-] \quad (16-4)$$

If the concentrations of Ag^+ and OAc^- in a saturated solution are measured, the K_{sp} value can be determined. Hence, measure a fixed volume of silver nitrate (AgNO_3) and sodium acetate (CH_3COONa) solutions of known concentrations, which satisfy the condition $Q > K_{sp}$. Then mix the two solutions to precipitate $\text{AgOAc}(s)$ and obtain

a saturated AgOAc solution. After filtering out the precipitate, the equilibrium concentration of Ag^+ in solution is determined by titration with SCN^- using Fe^{3+} as an indicator. Ag^+ reacts completely with SCN^- to form a white AgSCN precipitate, as shown in equation 16-5. Beyond the equivalence point, a small excess of SCN^- will react with Fe^{3+} to form red FeSCN^{2+} complex ions (16-6), signaling the end-point of the titration. This titration gives the concentration of Ag^+ , while the concentration of acetate ion can be determined by stoichiometric calculations.



In general, K_{sp} is independent of the concentration of ions or pH value. However, K_{sp} changes with temperatures. The K_{sp} values of most substances increase with increasing temperature. Since higher temperature supplies more energy to break the crystal lattice of the precipitate, the solubility is increased. For example, PbCl_2 and Ag_2SO_4 are insoluble in cold water but soluble in hot water. In this experiment, we will determine the K_{sp} values of silver acetate at different temperatures.

Apparatus

Stirrer/hot plate, magnetic stirring bar, buret (25 mL), graduated pipet (10 mL), pipet filler, Erlenmeyer flasks (125 mL, 4), beakers (100 mL, 4), thermometer, Styrofoam cup, funnel, filter paper, supporting ring, and latex gloves.

Chemicals

0.050 M Potassium thiocyanate, KSCN

0.20 M Silver nitrate, AgNO_3

0.30 M Sodium acetate, CH_3COONa

0.17 M Fe(III) indicator: ammonium ferric sulfate dodecahydrate in 6 M HNO_3 ,
 $\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$

Procedure

I. K_{sp} of silver acetate at room temperature

1. Wash clean and oven dry four 100 mL beakers and two funnels in advance.
2. Measure accurately 10.0 mL of 0.20 M AgNO_3 and 15.0 mL of 0.30 M NaOAc solutions, and add them to a 100 mL beaker. Mix and stir for 20 minutes.

Note: Refer to the experimental skills videos to learn how to use a stirrer/hot plate.

3. Record the temperature of the solution and then filter it. The filtrate is a saturated AgOAc solution.

Note 1: Refer to the experimental skills videos to learn how to perform gravity filtration.

Note 2: Do not wet the filter paper used in this step to avoid affecting the concentration of saturated solution.

4. Use a 10 mL graduated pipet and a pipet filler to transfer accurately 5.0 mL of saturated AgOAc solution to an Erlenmeyer flask. Add 1 mL of Fe³⁺ indicator and perform a preliminary “rough” titration.

Note: Practice using the graduated pipet and pipet filler with water first. Refer to the experimental skills videos to learn how to use graduated pipet and pipet filler.

5. Wash a 25 mL buret clean and rinse it twice with ca. 5 mL of 0.050 M KSCN standard solution each time. Fill the buret with 0.050 M KSCN(aq) and record the initial volume (V_i) to 0.01 mL.

Note 1: Use a clean and dry 100 mL beaker to take ca. 30 mL 0.050 M KSCN(aq).

Note 2: Expel air from the buret tip. Refer to the experimental skills videos to learn how to use a buret.

6. Perform titration with 0.050 M KSCN(aq) (a white AgSCN precipitate is formed) until the solution turns orange-red (FeSCN²⁺). The end-point is reached when the color persists for 15 s of swirling. Read and record the final volume (V_f) to 0.01 mL. Use the result of this “rough” titration to estimate the volume of KSCN(aq) needed for the precise titration of the silver acetate solution in the following procedures.
7. Dispose the solution in the Erlenmeyer flask to the indicated silver containing waste container. Brush and wash the flask with tap water and rinse with DI water. Measure accurately 10.0 mL of saturated AgOAc solution and transfer it to the clean flask. Add 1 mL of Fe³⁺ indicator and perform a second and precise titration.

II. K_{sp} of silver acetate at low temperature

8. Repeat steps 2~3, but place the beaker with solution in an ice-water bath and stir it for 20 min until the equilibrium is reached. Measure and record the equilibrium temperature; then filter the solution immediately.

Note: During the filtration, the remaining solution in the beaker should be kept in the ice-water bath to prevent the dissolution of the AgOAc precipitate caused by the temperature change, which would change the concentration of Ag⁺ ions.

9. Repeat steps 4~7 with the filtrate obtained in step 8 to determine the titration volume of KSCN.
10. Both the liquid waste and the precipitate of this experiment contain the noble metal Ag. Recycle them to the indicated silver containing waste container.

References

1. Skoog, D. A.; West, D. M. *Fundamentals of Analytical Chemistry*; 3rd ed., Holt, Rinehart & Winston, Inc.: New York, 1976; pp180-181.
2. Harris, D. C. *Quantitative Chemical Analysis*, 5th ed.; W. H. Freeman and Company: New York, 1999; p 164.