LABORATORY EXPERIMENT 6

PRECIPITATION TITRATION WITH SILVER NITRATE.

The $AgNO_3$ solution (~0.02 M) needs to be standardized using NaCl (FW 58.44) as a primary standard. You will perform standardization using Fajans method with adsorption indicator and using Mohr method with chromate indicator. Both titrations are to be done in triplicate.

I. Standardization of $AgNO_3$ solution

1. The Determination of Chloride by Titration with an Adsorption Indicator

Discussion

In this titration, the anionic adsorption indicator dichlorofluorescein is used to locate the end point. With the first excess of titrant, the indicator is incorporated into the counter-ion layer surrounding the silver chloride and imparts color to the solid. To obtain a satisfactory color change, it is desirable to maintain the particles of silver chloride in the colloidal state.

INDICATOR

Dichlorofluorescein indicator (sufficient for several hundred titrations). Dissolve 0.2 g of dichlorofluorescein in a solution prepared by mixing 75 mL of ethanol and 25 mL of water.

PROCEDURE

Dry the NaCl sample at 110°C for about 1 hr; allow it to return to room temperature in a desiccator (this will we done for you). If 0.02 M $AgNO_3$ is to be used, weigh a 0.15-0.20 g NaCl sample (exact mass!) into a 500-mL volumetric flask, and take 25-mL aliquots for titration. To each, add 5 drops of indicator. Titrate with $AgNO_3$ to the first permanent pink color of silver dichlorofluoresceinate.

Note:

Colloidal AgCl is sensitive to photodecomposition, particularly in the presence of the indicator; attempts to perform the titration in ***direct sunlight*** will fail. If photodecomposition appears to be a problem, establish the approximate end point with a rough preliminary titration, and use this information to estimate the volumes of AgNO3 needed for the other samples. For each subsequent sample, add the indicator only after most of the AgNO3 has been added, and then complete the titration without delay.

2. The Determination of Chloride by the Mohr Method

Discussion

The Mohr method uses chromate ion as an indicator in the titration of chloride ion with silver nitrate. The first excess of titrant results in the formation of a red silver chromate precipitate, which signals the end point.

INDICATOR

Sodium chromate, 5% Indicator

PROCEDURE

Introduce several drops of Na_2CrO_4 solution, and titrate to the first permanent appearance of red Ag_2CrO4.

Calculations:

From the volume of silver nitrate solution used fopr titration, calculate the molarity of $AgNO_3$. As usual,

$$m_{NaCl} = \frac{FW_{NaCl} \times C_{AgNO_3} \times V_{AgNO_3}}{1000} \times \frac{V_{total}}{V_{aliquote}}$$

This equation should be solved for C_{AgNO3}

$$C_{AgNO_3} = \frac{m_{NaCl} \times 1000 \times V_{aliquote}}{FW_{NaCl} \times V_{AgNO_3} \times V_{total}}$$

Standard silver nitrate solution can be used for various titrations.

II. Fast Determination of Sodium Chloride in Ketchup

Mixtures of halides can be titrated with $AgNO3$ solution as described in the textbook. In this experiment, you will use the apparatus in the textbook to monitor the activity of $Ag+$ as the titration proceeds. The theory of the potentiometric measurement is described in the textbook.

A 0.4 M bisulfate buffer (mixture of NaHSO4 and H2SO4, pH 2) will be available in the lab.

Weigh 0.3-0.4 g of ketchup, transfer it into a beaker for titration, add 30-40 mL of water, 2 mL of bisulfate buffer (see above), shake well, and titrate with $AgNO_3$ to the same chloride end point as in the previous determination. Calculate the percent of NaCl in ketchup.

For other similar samples (tomato juice, or other vegetable juice, salsa, etc.) adjust the amount you way accordingly. There is no need to use analytical balances in this case: you may use technical top-load balances with uncertainty 0.001 g or 0.001 g. Therefore, you can weigh the sample directly into a titration beaker.

$$m_{NaCl} = \frac{FW_{NaCl} \times C_{AgNO_3} \times (V_{AgNO_3})}{1000}$$

$$\%\%(NaCl) = \frac{m_{NaCl} \times 100\%}{m_{ketchup}}$$