Ministry 0f Higher Education and scientific Research

Al-Mustansiriya University

Collage of Science

Department of Chemistry

Practice Volumetric Chemical Analysis

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2// Precipitation titration:-

Volumetric methods based upon the formation of sparingly soluble precipitate are called **precipitation titration**; different titrimetric procedures that take place in solution were discussed. A special type of titrimetric procedures involves the formation of precipitates during the course of a titration. The titrant reacts with the analyte forming an insoluble material and the titration continues till the very last amount of analyte is consumed. The first drop of titrant in excess will react with an indicator resulting in a color change and announcing the termination of the titration. Precipitation titration is a very important; because it is a perfect method for determine halogens and some metal ions. There are three kinds (types) of indicators used in precipitation titration, the first used K_2CrO_4 (mohr, formation color precipitation method), the second used fluorescein indicator (fajan method), and the third used Fe^{+3} ion as indicator (volhard method back- titration formation color complex method).

Experiment No.(4):- Preparation and standardization of 0.1 N $AgNO_3$ solution with sodium chloride (Mohr Method)

Theory:-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.

$$Ag^+ + X^- \longrightarrow AgX + NaNO_3$$

$$AgNO_3 + NaCl \longrightarrow AgCl + NaNO_3$$

$$2AgNO_3 + K_2CrO_4 \longrightarrow AgCrO_4 + 2KNO_3$$

yellow — red ppt.

Procedure:-

1-Standardization of silver nitrate solution:-Sodium chloride has a relative molecular mass of 58.44. A 0.05 M solution is prepared by weighing out 0.29 g of the pure dry salt and dissolving it in 50 mL of water in a volumetric flask.

2. Preparation approximately 0.1 N $AgNO_3$: calculate the Wt. in 50 ml of $AgNO_3$ from:-

$$N = \frac{Wt.}{Eq.Wt.} * \frac{1000}{V\ (ml)}$$

$$0.05 = \frac{Wt.}{169} * \frac{1000}{50}$$

Weigh X g of $AgNO_3$ in dry and clean beaker then transfer to 50 ml volumetric flask and complete the volume to the mark with D.W.

3. Transfer 5 ml volume of the(0.02 N NaCl) solution, with a pipette, to a conical flask. Capacity 300 ml.

4. Add 4-5 drops of potassium chromate indicator K_2CrO_4 to this solution.

5. Add (0.05 N $AgNO_3$) from the burette gradually with continuous swirling of the solution in the conical flask and near the end point, $AgNO_3$ is added drop by drop. Continue the addition of $AgNO_3$ until appears red Precipitate.

6. Repeat step 2 three time to take the average of volume (V).

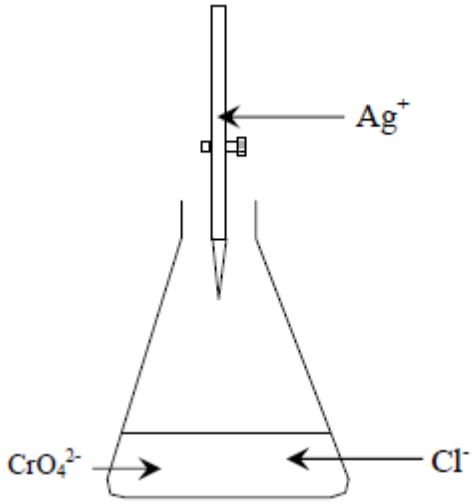
7. Find the error of titration process by Transfer 5 ml volume of the D.W, with a pipette, to a conical flask. capacity 300 ml, add 4-5 drops of potassium chromate indicator K_2CrO_4 to this solution, Add (0.05 N $AgNO_3$) from the burette gradually with continuous swirling of the solution in the conical flask and near the end point, $AgNO_3$ is added drop by drop. Continue the addition of $AgNO_3$ until upper red Precipitate appears, Recorded the volume (V1).

Calculations:-Calculate the normality of Cl^-:-

$$(N * V)_{NaCl} = (N * V)_{AgNO3}$$

$$(N * 5) = (0.05 * V - V1)$$

$$conc.(gm/L)_{Cl^-} = N * Eq.Wt$$



Discussion:-

1. What is the red precipitate?
2. What is the name of indicator?
3. What is the error of titration process?
4. What the second name of this method?
5. Why AgCl Precipitate first than Ag_2CrO_4?
6. Why $AgNO_3$ solution must be standardized first?

Experiment No.(5):- Determination of Chloride by Fajan's method (Adsorption indictors)

Theory:-In Fajan method, the end point is detected with a dye that imparts a distinctive color to the silver chloride precipitate. Dichlorofluorescein or Fluorescein (HIn) is commonly used as indicator. As the surface equivalence point is approached and passed, silver ion will become the primary adsorbed the surface of precipitate. The negatively charged dichlorofluorescein or Fluorescein anion (In^-) will then on displaces the nitrate ion to become the countering. On being adsorbed, its electronic changes so that it reflects reddish /pink light rather than yellow-green, this signals the end point.

$$AgCl: Ag^+/NO_3^-(s) + HIn\ (yellow) \rightarrow AgCl:Ag^+/In^-\ (pink) + H^+ + NO_3^-$$

Procedure:-

1. Transfer 25 ml volume of the NaCl or KCl solution, with a pipette, to a conical flask.

2. Add 5-10 drops Dichlorofluorescein or Fluorescein and 0.1 gm dextrin solution. The color of solution becomes yellow-green.

3. Add 0.0.5 M $AgNO_3$ from the burette gradually with continuous swirling of the solution in the conical flask and near the end point, $AgNO_3$ is added drop by drop. Continue the addition of $AgNO_3$ until appears reddish Precipitate

Calculations:-Calculate the molarity of chloride:-

$$(M * V)\mathbf{Cl^-} = (M * V)\ \mathbf{AgNO_3}$$

Calculate PCl:- $PCl = - Log [Cl^-]$

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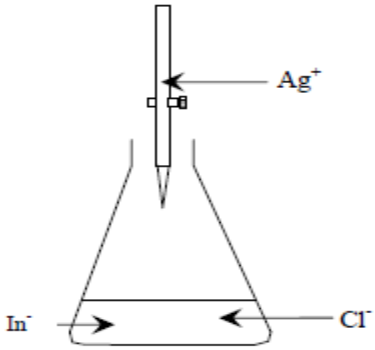

Discussion:-

1. What is the HIn?

2. Define adsorbed process?

3. What is the adsorbed indicator?

4. Why dextrin solution is added?

5. What is the name of precipitation involved Ag?

6. Why the used indicator is called Fluorescein indicator?