C241

NAME:_____ BATCH:_____

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ESTIMATION OF TOTAL MANGENESE IN MANGANESE ORE (PYROLUSITE) (REDOX TITRATION)

AIM

A method for analysis of metal ores: (eg. Mn^{2+} in pyrolusite ore using oxidation-reduction titration).

THEORY

Redox titrations are also called as oxidation-reduction titration between an oxidising agent and a reducing agent. It can be determined volumetrically by using a redox indicator or by potentiometric titration using instrument. However, some redox titrations do not require indicator, for example, in the redox titration of $KMnO_4$ and ferric salt, purple colored solution of MnO_4^- ions (oxidizing agent) changes in to colorless Mn^{+2} ions at the equivalence point.

In this experiment you have to estimate total manganese content in pyrolusite ore by redox titration method. Pyrolusite is the ore of manganese containing Mn as MnO_2 with some MnO and Fe_2O_3. In this estimation, permanganate will oxidize $C_2O_4^{2-}$ in acid medium quantitatively as per its higher standard reduction potential.

$$MnO_2 + 4H^+ + 2e^- \rightleftharpoons Mn2^+ + 2H_2O \qquad E^0 = 1.23\ V$$

$$2CO_2 + 2e^- \rightleftharpoons C_2O_4^{2-} \qquad E^0 = -0.49\ V$$

Since oxalate reacts very slowly at room temperature so the solutions are titrated hot to make the analysis in practical.

MATERIALS REQUIRED

Potassium permanganate, potassium oxalate monohydrate, sulphuric acid (6N), pyrolusite ore, iron ore (iron salt), burette (50 mL), conical flask (250 mL), pipette (25 mL), measuring cylinder, volumetric flask, funnel, watch glass, hot plate, analytical balance, weighing boat or paper, amber colored bottle, dropper, distilled water etc.

PROCEDURE

1. Standardization of $KMnO_4$ solution

1. Prepare primary standard solution of $K_2C_2O_4.H_2O$ (~0.1N) solution ($Na_2C_2O_4$ can also be used).
2. Standardize the unknown permanganate solution as secondary standard oxidant using a primary standard reductant.

Titration procedure: Pipette out 25 mL aliquot of standard $K_2C_2O_4$ in to a conical flask. Then add 50 mL of 6N H_2SO_4 to the oxalate sample in the conical flask. Heat the acidified oxalate solution to about 70-80°C. Titrate the hot oxalate solution with the $KMnO_4$ solution until the appearance of a faint pink color. Note the final burette reading and repeat the titration to get three concurrent results. Calculate the strength of $KMnO_4$ solution.

$$MnO_4^- + 4H^+ + 5e^- \rightleftharpoons Mn^{2+} + 4H_2O \qquad E^0 = 1.51\ V$$

$$2CO_2 + 2e^- \rightleftharpoons C_2O_4^{2-} \qquad E^0 = -0.49\ V$$

Table for calculation of strength of $KMnO_4$

S.N.	Initial burette reading (mL)	Final burette reading (mL)	Vol. of $KMnO_4$ used

2. Estimation of Mn^{2+} in Manganese ore
(Back titration method)

1. Weigh ~0.2 gm of pulverized pyrolusite sample using analytical balance and record the exact mass.
2. Transfer the mass into a conical flask and add 33 mL of 6N H_2SO_4 followed by the addition of 30 mL of standard (N/10) $K_2C_2O_4.2H_2O$ solution using pipette.
3. Cover the mouth of the flask with short stem funnel and heat until all the black particles are dissolved.
4. Cool the solution to around 70°C and back titrate the excess $K_2C_2O_4.2H_2O$ in the mixture against standard $KMnO_4$ solution until the appearance of a faint pink color.

S.N.	Initial burette reading (mL)	Final burette reading (mL)	Vol. of $KMnO_4$ used

Results and observation

1. Strength of $KMnO_4$ solution =
2. Strength of $K_2C_2O_4.H_2O$ solution =
3. Mn% in pyrolusite ore =
4. Estimate iron in a given salt by direct titration method (hint: titrate directly with $KMnO_4$, do not add oxalate solution)

$$Fe^{3+} + 2e^- \rightleftharpoons Fe^{2+} \qquad E^0 = -0.77\ V$$