Chemistry (P.G.)
[CBCS]
M.Sc. First Semester End Examination-2023
(Regular & Supplementary Paper)
PAPER- CEM-195 (Practical)

Full Marks: 50 ***Time: 06 Hrs***

The figures in the right-hand margin indicate marks.
Candidates are required to give their answers in their own words as far as practicable.
Illustrate the answers wherever necessary.

1. Determine the amount of Fe^{3+} and Cu^{2+} ions present in the supplied sample solution.

(i) Tables	08
(ii) Results	16
(iii) Calculations	06
(iv) Conclusion	02

2. Preparation of potassium tris oxalato chromate(III) trihydrate. Mention

(i)	Chemical formula and	03
(ii)	Yield	05

3.	Viva-Voce	07
4.	Laboratory note book	03

❖ **Process for determination of the composition of Dolomite**

a) **Preparation of 250 mL (N/20) standard $K_2Cr_2O_7$ solution:** A precise weight is to be taken and dissolved in distilled water and made up to the mark in a 250 mL volumetric flask. Mention the weight taken upto 4 decimal places.

b) **Standardization of $Na_2S_2O_3$ solution by standard (N/20) K2Cr2O7 solution:** 25 mL of standard $K_2Cr_2O_7$ solution is pipetted out in a 500 mL conical flask. Add 2 g sodium carbonate followed by 25 mL 4N H_2SO_4. Swirl well the solution and wait for 2 minutes. Add 2 g KI, cover the flask with watch glass and keep it in dark for 5 minutes. Wash the covering watch glass and inner surface of conical flask with water. Dilute with 150 mL water. Titrate with $Na_2S_2O_3$ solution to straw yellow.

colour. Add 2 mL of starch solution, as the solution turns deep blue colour. Continue titration with standard $Na_2S_2O_3$ solution till the blue colour of the solution disappears and light green colour solution persists. Record the titre value.

c) **Estimation of Cu(II) ions:** 25 mL of supplied sample solution is pipetted out in a 250 mL conical flask. Neutralize by 1:1 NH_4OH till a faint bluish white turbidity appear. Add 2 g NH_4HF_2 to dissolve the precipitate, followed by 2 g of KI. Dilute the solution to 100 mL. Titrate with standard $Na_2S_2O_3$ solution to straw yellow colour. Add 2 mL of starch solution, as the solution turns deep blue colour. Continue titration with standard $Na_2S_2O_3$ solution till the blue colour of the solution faded away. Add 10 mL 1% NH_4SCN, shake well and continue

the titration till blue colour completely discharge and creamy white solution results. Record the titre value.

d) **Estimation of Fe(III) ions:** 25 mL of stock solution is pipetted out in a 500 mL beaker and diluted to 100 mL with water. Add 2 g of NH_4Cl, heated to boiling. Add NH_4OH dropwise till smell of NH_4OH persists. Reddish brown precipitate appears and allow to settle down on a hot asbestos board. Filter hot through Whatman 41 filter paper. Wash with 1% NH_4Cl aqueous solution containing few drops of NH_4OH. Dissolve the precipitate in minimum volume of hot (1:1) HCl, followed by hot water and collect in the same beaker where initial precipitation was carried out. Re-precipitation process carried out. Reduce the solution by Al foil and heat the solution carefully (avoid boiling). Shake the solution continuously until the yellow colour discharged. Cool the solution and add 5 mL Phosphoric acid followed by few drops of BDS indicator. Titrate with standard (N/20) $K_2Cr_2O_7$ solution till colour changes from green to violet. Record the titre value

Procedure of preparation of potassium tris oxalate chromate(III) trihydrate

2.8 gm of potassium oxalate monohydrate and 5.5 gm oxalic acid dihydrate was taken in a beaker and then 110 to 120 ml water was added to it. 1.9 gm solid $K_2Cr_2O_7$ was added to it in small portion with constant stirring. The solution was concentrated nearly to dryness on cooling deep green shiny crystal of potassium trisoxalato chromate(III) form. The solid was filtered by pressing between filter paper.