Ref. No.: Ex/UG/Sc./Core/Chem/PR/05/2/21/2019

B. Sc. Chemistry 2nd Year 1st Sem (PRACTICAL) EXAMINATION, 2019

CHEMISTRY (Practical)

Full Marks – 30 Time- 10:00 am onwards

Q. 1. Estimation of Fe(III)and Ca(III) in a given mixture 20
Q. 2. Viva & Note book 10

Suggestions for Q.1.

(a) Transfer quantitatively the given sample into a 250 ml volumetric flask and adjust the volume upto the mark. Shake well to make it homogenous.

(b) Prepare a 250 ml 0.1 N standard oxalic acid solution.

(c) Collect the supplied $KMnO_4$ solution.

(d) **Standardization of the $KMnO_4$ solution**.
Pipette out 25 ml oxalic acid solution into a 250 ml volumetric flask. Add 150 ml 2(N) H_2SO_4 solution. Heat the mixture to ~ 70°C. Titrate this solution with the supplied $KMnO_4$ solution adding it from a burette until a faint pink colour persists for at least 30 seconds. Note and record the burette reading.

(e) **Separation of Fe(III) from the mixture**:
Pipette out 25 ml of the Fe(III)-Ca(II) stock solution in a 500 ml beaker marked IRON. Add 2 gm. NH_4Cl and 150 ml distilled water. Heat the solution to boiling and keep it on a hot asbestos board. Add 1:1 NH_3 drop wise to turbidity with continuous stirring till the solution becomes faintly ammoniacal (pH ~ 9.0). A brown precipitate appears. Remove the burner and cover the beaker with a watch glass. Allow it to settle for the next 5 minutes. Filter using Whatman 41 filter paper. Wash the beaker and the precipitate 4 to 5 times with hot 1% NH_4Cl solution containing 1-2 drops of liquor ammonia (in 50 ml). Filtrate and washings are to be collected in a 500 ml beaker marked CALCIUM.
Reprecipitation: Dissolve the precipitate on the filter paper with 6 N HCl (25 ml conc. HCl and 25 ml distilled water) and collect it in the beaker marked IRON. Adjust volume to 150 ml and re-precipitate Fe(III) as earlier. The filtrate and washings are to be collected in the beaker marked CALCIUM.

(f) **Estimation of Iron**: Dissolve the precipitate in a minimum quantity of hot 6 N HCl (described above) until the filter paper has no yellow stains on it. Collect the solution and washings in the beaker marked IRON. Heat the solution to boiling and reduce with $SnCl_2$ adding drop wise with stirring till the solution is colourless; add 1 drop excess. Add 10 ml $HgCl_2$ solution all at once to get a silky white precipitate. Diluted to 150 ml with distilled water and add 25 ml Zimmermann Reinhardt solution and 10 ml syrupy H_3PO_4. Titrate the mixture immediately with standard $KMnO_4$ solution till the colour turns light pink.

(g) **Estimation of Calcium**: The volume of the filtrate containing Ca(II) is reduced to 150 ml. Add 2-3 drops of methyl red indicator and 6 N HCl drop wise till the solution turns red. Heat the solution to boiling and add 10 ml 5% $(NH_4)_2(COO)_2$ solution. Add 1:1 NH_3 drop wise with stirring till the solution turns orange or yellow. Cover the mouth of the beaker with a watch glass and allow the precipitate to settle for the next 15 minutes. Filter the Calcium oxalate precipitate using Whatman 42 filter paper. Wash the precipitate using hot 1% $(NH_4)_2(COO)_2$ solution and then with boiling water till washings are free of Cl^-. Reject the filtrate. Pierce the filter paper with a glass rod and wash down the precipitate to the beaker marked CALCIUM. Wash the filter paper with 4 N H_2SO_4 and hot water till all Calcium oxalate has been transferred to the beaker. Adjust volume to ~200 ml maintaining acidity at > 2 N with respect to H_2SO_4. Heat the solution to ~ 70°C and titrate against standard $KMnO_4$ solution to a faint pink end point.