Jadavpur University
B. Sc.(H) Final Year Chemistry Second Semester
Analytical Chemistry Practical Examination-2018

Full Marks – 30, Time – 11-00 am - 5-00 pm (1st Day); 11.00 am – 2.00 pm (2nd Day)

Q. 1. Estimation of Fe^{3+} and Ca^{2+} in a given mixture 18

Hints:

(a) Transfer quantitatively the given sample into a 250 ml volumetric flask and make up the volume.

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

(c) Standardize the supplied $KMnO_4$ solution.

(d) Prepare 0.01 M standard Na_2EDTA solution.

(e) Separation and Estimation of Fe^{3+} from the mixture:

Pipette out 25 ml of aliquot from the supplied solution in a 500 ml beaker. Dilute to 100 ml with distilled water, add ~ 1.5 gm NH_4Cl and heat to nearly boiling. Then add 1:1 NH_3 solution drop-by-drop to precipitate $Fe(OH)_3$ completely. Settle the precipitate and filter through Whatmann No. 41 filter paper. Wash the precipitate 3-4 times with 0.1% NH_4NO_3 solution. Dissolve the precipitate in a minimum vol of HCl (1:1) and reprecipitate. Filter and wash the precipitate as done above. The *filtrate from both experiments is to be kept aside for estimation of Calcium.* Dissolve the precipitate into a minimum vol of 1:1 HCl and dilute by adding 50 ml water. Heat this mixture nearly to boiling and reduce Fe(III) by $SnCl_2$ solution. Cool the solution and add 10 ml $HgCl_2$ solution at once. Dilute this solution to 200 ml, add 25 ml Z-R solution and 10 ml syrupy phosphoric acid. Titrate the mixture immediately with a standard $KMnO_4$ solution till the colour turns light pink by one drop at the end point.

Report the volume of titration in Tabular form and calculate total amount of iron in the given mixture.

(e) Estimation of Calcium(II) in the mixture :

Filtrate collected from previous experiment is to be heated to reduce its volume to 100 ml (approx). Add 2-3 drops of methyl red indicator and then add 6(N) HCl solution drop wise with stirring until the solution turns red. Heat the solution nearly to boiling and add 10 ml 5% ammonium oxalate solution. Add 1:1 aq-NH_3 dropwise with stirring till the solution turns orange-yellow. Add 2 ml ammonium oxalate solution. Allow the solution to stand for 15 min. Filter the precipitate of Calcium oxalate through Whatmann No. 42 filter paper. Wash the precipitate by 0.1% ammonium oxalate solution thrice followed by boiling water till the washing is free from Cl^- ion (test with $AgNO_3$ solution in nitric acid medium). Reject the filtrate. Pierce the filter paper with the tip of a glass rod and wash down the precipitate to a beaker. Wash the filter paper with 4(N) H_2SO_4 and hot water, adjust the final volume to 200 ml and maintain ~2(N) w.r.t. H_2SO_4. Heat the solution to ~70 - 80°C and titrate against a standard $KMnO_4$ solution to a light pink end point. Report the volume of titration in Tabular form and calculate total amount of calcium in the given mixture.

Q.2. Estimate total hardness of water complexometrically following usual procedure. Report hardness of water in ppm. 07

Q.3. Viva & Note Book 05