Ref. No. Ex/FC/2/VII/PR/77/2017

JADAVPUR UNIVERSITY

B. Sc. (H) Final Year Chemistry Second Semester

Practical Examination – 2017

Analytical Chemistry

Full Marks – 30: Time : 1st Day (11.00 a.m. – 5.00 p.m.)
2nd Day (11.00 a.m. – 2.00 p.m.)

Q. 1. Estimation of Fe(III)and Cr(III) in a given mixture	18
Q. 2. Estimate hardness of supplied water sample by complexometric titration	07
Q. 3. Viva & Note book	05

Hints: Q.1.

(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 $K_2Cr_2O_7$ solution

(c) Standardisation of Mohr Salt
Pipette out 25 ml Mohr Solution, add 50 ml 2(N) H_2SO_4, 20 ml H_3PO_4, 3-4 drops BDS indicator and titrate against a standard $K_2Cr_2O_7$ solution. Colour changes to blue-violet at the end point.

(d) Separation of Fe(III) from the mixture:
Pipette out 25 ml of stock solution in a 500 ml beaker. Add $(NH_4)_2CO_3$ till turbidity appears followed by 1 g Na_2O_2 (pinch wise) with continuous stirring. The solution is to be heated to decompose excess peroxide for 10 min. The mass is diluted to 100 ml and allowed to settle. Filter using Whatman 41 filter paper and wash with 0.1% NaOH solution till free from chromate. One drop of the filtrate is to be tested by acidifying with acetic acid and then adding a drop of $AgNO_3$ solution. The filtrate and the washings are collected in a conical flask. Re-precipitation is to be done as before.

(e) Estimation of Iron: The precipitate is dissolved in minimum quantity of hot (1:1) HCl, washed alternately with distilled water until the filter paper is colourless; 15 ml. of conc. HCl is added, heated almost to boiling, reduced with $SnCl_2$ solution, 10 ml. of $HgCl_2$ solution is added all at once to get a silky white precipitate. The solution is to be diluted to 150-200 ml with distilled water. 5 ml H_3PO_4 is to be added followed by the addition of 2-3 drops of BDS indicator. Titrate with standard $K_2Cr_2O_7$ solution, the first appearance of red-violet colouration indicates the end point of the titration.

(f) Estimation of Chromium: The volume of the filtrate containing $CrO_4^{=}$ is reduced to 50 ml. by evaporation (if necessary), cooled, acidified with 4(N) H_2SO_4 (colour changes from yellow to orange). A measured excess of standard Mohr's salt solution (50 ml) is added to discharge the dichromate colour, 5 ml. of H_3PO_4 and 2-3 drops of BDS indicator are then added and excess Mohr's salt is back titrated with standard $K_2Cr_2O_7$ solution. The first appearance of red-violet colouration indicates the end point of the titration.

Hints Q.2.

(a) Prepare 0.01 M Na_2EDTA solution in 250 ml volumetric flask.

(b) Pipette out 50 ml of the supplied water sample and dilute it to 100 ml with distilled water. Add 10 ml buffer (pH = 10) solution, 4-5 drops of EBT indicator and titrate with Na_2EDTA solution until the colour changes from wine red to blue. Perform a blank experiment. Do the blank titration following the same procedure.