NEW

Part–III 3-Tier

2016

CHEMISTRY

(Honours)

PAPER—VIII

(PRACTICAL)

Full Marks : 150

Time : 6 Hours a day (3 Days)

The figures in the right-hand margin indicate full marks.

Result must be recorded in tabular form as far as possible.

1. Estimate the amount of Fe^{3+} and Cu^{2+} ions quantitatively in the supplied solution marked 'V'. 30

2. Perform any one physical chemistry experiment from the supplied list of experiments.
   Marks are distributed into the following items :

Theory, Temperature recording, Representation of data and Tabulation, Calculation, Graph plotting (if necessary) and Results. 60

3. Estimate the the amount of Fe_2O_3 in the Supplied Portland Cement. 30

4. Laboratory Note Book. 15

5. Viva-Voce. 15

[Procedure]

1. Estimation of Fe^{3+} and Cu^{2+} ions in the supplied solution :

(i) ***Preparation of stock solution :***

Carefully open the cap of the sample bottle and then transfer the supplied solution quantitatively into a 250 ml volumetric flask. Finally make the volume up to the mark using distilled water.

(ii) ***Preparation of 250 ml standard (N/20) $K_2Cr_2O_7$ solution :***

Weigh out accurately 0·6125gm $K_2Cr_2O_7$ (A.R. grade) and dissolve it by distilled water in a 250ml volumetric flask.

(iii) ***Standardization of given Sodium thiosulphate solution :***

Pipette out 25 ml of standard $\left(\frac{N}{20}\right)$ $K_2Cr_2O_7$ solution into a 500ml conical flask. Add 25ml of 4 (N) H_2SO_4, 2gm of KI, close the mouth of the conical flask with a watch glass and keep the flask with its contents in dark for 2-3 minutes. Wash down the watch glass and sides of the flask with distilled water. Dilute to 200ml and titrate immediately with sodium thiosulphate solution running from a burette until the brown colour fades to straw yellow. Add 2ml of 1% freshly Prepared Starch Solution and Shake to obtain a deep blue colour. Then titrate with thiosulphate solution very carefully until the colour changes sharply from intense blue to bright green with one drop. Record the titre of the thiosulfate solution and calculate its strength.

(iv) ***Estimation of Cu^{2+} ion :***

Pipette out 25 ml of stock solution into a 500 ml conical flask, neutralise with (1:1) NH_3 to obtain a permanent turbidity (avoid excess NH_3) and dissolve the same by adding ~ 2 gm of NH_4HF_2. Add 2 gm of KI, dilute to 100 ml and titrate immediately with standard thio sulphate solution until brown colour

fades to straw yellow. Add 2 m*l* of 1% starch solution and continue titration until colour fades to pale blue. Then add 1gm of solid NH_4SCN, shake and titrate until pale blue colour just disappears to give milky white solution. Note the titre value to calculate the total amount of Cu^{2+} ion present in the supplied sample.

(v) ***Estimation of Fe^{3+} ion :***

Pipette out 25 ml of the stock solution into a 500 ml beaker. Dilute the solution to about 100 ml with distilled water, add 1gm NH_4Cl and warm a little. Add dropwise 1 : 1 aqueous ammonia solution with constant stirring till the smell of ammonia persist. Allow the precipitate to settle for 5 min. and then filter through a what man filter paper no. 41. Wash the precipitate twice with washing liquid, 1% aqueous NH_4Cl solution containing few drops of NH_3. Dissolve the precipitate in minimum volume of hot (1:1) HC*l* and hot distilled water, successively. Reprecipitate Fe^{3+} ion quantitatively with (1:1) aqueous ammonia as mentioned earlier and allow to stand for settling down the precipitate. Refilter the precipitate through the same filter paper and wash as before. Dissolve the precipitate in 50 ml of (1:1) hot HCl and finally wash with hot distilled water until the filter paper becomes

colourless. Heat the solution to about 80°C then add small pieces of AR grade Al-foil stepwise to reduce Fe(III) quantitatively to Fe(II), swirl the solution till all the Al-pieces gets dissolved giving rise a clear solution. Cool the solution to room temperature and dilute to 150 ml with distilled water. Then add 5 ml of syrupy H_3PO_4, 4-5 drops BaDS indicator and titrate the solution with the standard (N/20) $K_2Cr_2O_7$ solution to a reddish-violet end point. Record the titre value to calculate the total amount of iron present in the supplied sample.

[N.B. : 1000 ml 1(N)$K_2Cr_2O_7$ ≡ 55.85 of Fe^{3+} and 1000ml 1(N) $Na_2S_2O_3$ ≡ 63·55 gm of Cu^{2+}]

3. Estimation of Fe_2O_3 in Portland Cement :

(i) Preparation of 250 ml standard (N/20) $K_2Cr_2O_7$ solution:

Weight out accurately 0·6125gm of $K_2Cr_2O_7$ (A.R. grade) and dissolve it by distilled water in a 250 ml volumetric flask.

(ii) Estimation of Fe_2O_3 in Portland Cement :

Transfer the supplied Cement Sample quantitatively into a 500 ml beaker and dissolve it in 50 ml distilled

water. Add 10 ml Conc. HCl by stirring with a glass rod. If the Sample does not dissolve, heat the mixture and after Complete dissolution, again heat the mixture almost to boiling. Then reduce it by $SnCl_2$ until the yellow solution is just colourless. Cool the mixture under the tap. Add 10 ml of $HgCl_2$ Solution, Stir and allow to stand for 2-3 minutes, until slight silky precipitate appears. Add 5 ml of syrupy H_3PO_4, followed by 2-3 drops BaDs indicator. Then titrate it with a standard (N/20) $K_2Cr_2O_7$ solution until first red-violet colour appears. Note the titre value to calculate the amount of Fe_2O_3 in supplied portland cement sample.
