OLD

Part-III 3-Tier

2017

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 total hardness of water in the supplied water sample. 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 0.1(N) $K_2Cr_2O_7$ solution :***

   Weigh out accurately 1·2257gm $K_2Cr_2O_7$ (A.R. grade) and dissolve it by distilled water in a 250ml volumetric flask.

(iii) *Standardization of given Mohr salt solution :*

Pipette out an aliquot of 25 ml Mohr's salt solution into a 500ml conical flask dilute to 150 ml with distilled water. Add 25ml of 4 (N) H_2SO_4, 2gm of NH_4HF_2 or 3 ml syrupy H_3PO_4 and 3-4 drops of BaDs indicator. Titrate the solution with standard 0.1(N) $K_2Cr_2O_7$ solution to violet end point.

(iv) *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) HCl 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 0.1(N) $K_2Cr_2O_7$ 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. : 1 ml 0.1(N)$K_2Cr_2O_7$ ≡ 0.005585 of Fe^{3+} and report the total amount of Fe in the supplied sample]

(v) ***Estimation of Cr^{6+} ion :***

Pipette out an aliquot of 25 ml from the stock solution into a 500 ml concical flask, add 25 ml of 4(N) H_2SO_4 and a measured excess volume (50 ml) of the standard Mohr's salt solution to discharge the orange colour of the solution. Add 5ml of H_3PO_4 or 2 gms of NH_4HF_2 and 3-4 drops of BaDs indicator and back titrate the excess Mohr's solution with same standard $K_2Cr_2O_7$ solution to violet end point. Calculate the total amount of Cr(VI) in the supplied solution from the difference in titre value.

[N.B. : 1 ml 0.1(N) Fe^{2+} solution ≡ 1ml 0.1(N) $K_2Cr_2O_7$ solution 0.001733 gm of Cr]

3. Estimation of total hardness of water :

(i) Preparation of 250 ml standard (M/100) $ZnSO_4$ solution:

Weigh out accurately 0.7188 g AR $ZnSO_4.7H_2O$ and dissolve it in a 250 ml volumetric flask using distilled water.

(ii) Standardization of supplied EDTA solution :

Pipette out 25 ml of standard (M/100) zinc sulphate solution in a 500 ml conical flask. Add 5 ml pH 10 buffer solution (NH_4Cl + NH_4OH) and dilute to 150 ml using distilled water. Shake the solution with 8-10 drops EBT indicator solution when the colour of the solution turns to wine red. Titrate the solution with the EDTA solution till the wine red colour turns blue.

(iii) Estimation of total hardness of water :

Take 50 ml of the supplied water sample in a 500 ml conical flask followed by the addition of 5 ml pH 10 buffer solution (NH_4Cl + NH_4OH) then dilute to 150 ml. Shake the solution with the addition of 8-10 drops

EBT indicator. Titrate the resulting wine red solution with the standardized (M/100) EDTA solution from burette until wine red colour turns to blue. Record the titre value and calculate the total hardness of water in ppm in terms of $CaCO_3$.
