NEW

Part-III 3-Tier

2015

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 $Cr_2O_7^{2-}$ 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 available oxygen in the supplied pyrolusite sample. 30

4. Laboratory Note Book. 15

5. Viva-Voce. 15

[Procedure]

1. Estimation of Fe^{3+} and $Cr_2O_7^{2-}$ 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 (N/20) $K_2Cr_2O_7$ solution :*** Weigh out accurately A.R. grade $K_2Cr_2O_7$ (Ca. 0.6128 g) and dissolve it by distilled water in a 250 volumetric flask.

to calculate the total amount of iron present in the supplied sample.

[N.B. : 1000 ml 1(N)$K_2Cr_2O_7$ ≡ 55.85 and 35.99 gm of Fe^{3+} and $Cr_2O_7^{2-}$, respectively]

3. Estimation of available oxygen in pyrolusite :

(i) Preparation of 250 ml standard (N/20) Oxalic acid solution:

Weight out accurately AR grade oxalic acid (Ca 0.7875 gm) and dissolve it in a 250 ml volumetric flask using distilled water.

(ii) Standardization of given $KMnO_4$ solution :

Pipette out 25 ml of standard (N/20) oxalic acid solution into a 500 ml conical flask. Add 50 ml 4(N) H_2SO_4 and dilute to 100 ml with distilled water. Heat the mixture on an asbestor board to ~80°C and titrate in the hot conditionwith $KMnO_4$ solution from a burette untill a faint pink colour lasting for ~30 mins is appeared.

(iii) Estimation of available oxygen in pyrolusite :

Transfer the supplied pyrolusite sample quantitatively into a 250 ml conical flask. Add 50 ml of standard (N/20) oxalic acid and 50 ml of 4(N) H_2SO_4 in quick

succession. Cover the mouth of the flask with a watch glass and heat the flask on an asbestor board at 80°C Untill all the black precipitate is dissolved (~ 20mins) Back titrate the excess oxalic acid with standard $KMnO_4$ solution and record the titre value to calculate the available oxygen in supplied pyrolusite sample.

[N.B. : Molecular weights of MnO_2 and O_2 are 86.94 and 32 gm, respectively]

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conical flask. Dilute the solution to about 100 r
distilled water and warm a little. Add dropwis
aqueous amonia solution with constant stirri
the smell of ammonia persist. Allow the prec
to settle down for 5 min and then filter thro
Whatman filter paper no 41. Wash the preci
twice with washing liquid, 1% aqueous NH_4Cl so
containing few drops of NH_3. Dissolve the preci
in minimum volume of hot 1 : 1 HCl and hot di
water, successively. Reprecipitate Fe^{3+}
quantitatively with 1 : 1 aqueous ammoni
mentioned earlier and allow to stand for settling
the precipitate. Refilter the precipitate through
same filter paper and wash the precipitate with
above mentioned washing liquid. Dissolve
precipitate in 50 ml of 1 : 1 hot HCl and finally
with hot distilled water untill the filter paper bec
colourless. Heat the solution to about 80°C then
small pieces of AR grade Al-foil stepwise to re
Fe(III) quantitatively to Fe(II), swirl the solution ti
the Al-pieces gets dissolved giving rise a
solution. Cool the solution to room temperature
dilute to 150 ml with distilled water. Add 5 ml of sy
H_3PO_4 and 4-5 drops BaDS indicator and titrate
solution with the standard (N/20) $K_2Cr_2O_7$ solu
to a reddish-violet end point. Record the titre v

(iii) ***Standardization of given Mohr's solution :***

Pipette out 25 ml of supplied Mohr's salt solution into a 500 ml conical flask. Add 50ml 4(N) H_2SO_4 and 5ml of surupy H_3PO_4 into the flask. Cool the resulting solution under tap if it becomes warmed and dilute to 150 ml using distilled water. Add adequate amount (4 - 5 drops). Barium diphenyl amine sulphonate (BaDS) indicator and titrate the solution with the standard (N/20) $K_2Cr_2O_7$ solution untill the reddish-violet colour appears.

(iv) ***Estimation of $Cr_2O_7^{2-}$ ion :***

Pipette out 25 ml of stock solution into a 500 ml conical flask. Add a measure excess (50 ml) of the standard Mohr's Salt solution, 50 ml of 4(N) H_2SO_4 and 5ml of surupy H_3PO_4 into the flask. Cool the resulting solution under tap if required and dilute to 150 ml using distilled water. Add 4-5 drops of BaDS indicator and back titrate excess Mohr's salt with the same standard (N/20) $K_2Cr_2O_7$ solution untill the appearance of reddish-violet colour. Note the titre avlue to calculate the total amount of $Cr_2O_7^{2-}$ ion present in the supplied sample.

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

Pipette out 25 ml of the stock solution into a 500 ml