NEW

Part–III 3-Tier

2018

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.

(Inorganic Chemistry)

1. (a) Estimate the total amount of Fe^{3+} and Ca^{2+} in the supplied solution quantitatively. 30

(b) Prepare Ammonium ferrous sulfate hexahydrate (Mohr's Salt) following given procedure. Repeat the yield of the dry product. 10

[Procedure]

1. (a) Estimation of Fe(III) and Ca(II) ions in the supplied solutions marked 'V' :

(i) Preparation of stock solution :

Transfer the supplied solution marked 'V' from the phial into a 250 ml volumetric flask quantitatively and make the volume up to the mark with distilled water.

(ii) Preparation of 250 ml standard (N/10) Oxalic acid solution.

(iii) Standardization of the given $KMnO_4$ solution :

Pipette out 25 ml standard Oxalic acid solutio in a 500 ml conical flask and dilute to 200 ml wi $2NH_2SO_4$.

Heat the mixture at about 70-80°C and titrate the hot solution slowly at first and then rapidly with $KMnO_4$ solution from burette. The end point will be marked by the appearance of very pale pink colour persisting for about 30 secs.

(v) Estimation of Fe(III)

Pipette out 25 ml of the stock solution into a 500 ml beaker, diluted to 100 ml with distilled water, add 1.0 gm of NH_4Cl, heat the solution nearly to boiling and add dropwise 1:1 NH_4OH with constant strring until ammonia smell persists. Allow the precipitate of $Fe(OH)_3$ to settle

and then filter it using Whatmann No. 41 filter paper and wash the precipitate with 1% NH_4Cl solution. Dissolve the precipitate in minimum volume of hot (1:1) HCl in the same beaker, reprecipitate and filter through the same filter paper and wash as before till free from calcium (to be tested with Ammonium Oxalate solution). The filtrate and the washings will be collected in a 500 ml beaker for estimation of calcium.

Dissolve the precipitate in the same beaker by hot (1:1) HCl, add 25 ml of conc. HCl, heat nearly to boiling and reduce Fe^{3+} to Fe^{2+} using small pieces of AR grade Al-foil stepwise. Dilute the solution to 200 ml with distilled water and add 25 ml Zimmermann Reinhardt solution. Finally titrate the mixture with standard $KMnO_4$ solution from the burette until a faint pink colour lasts 20 seconds. Record the titre value and calculate the amount of Fe(III) present in the supplied solution.

(vi) Estimation of Ca(II) :

Reduce the volume of the filtrate to about 200 ml and acidify the solution with dropwise addition of 4(N) HCl that can be understood by the colour change of methyl orange indicator from yellow to red.

Heat to boil the solution with addition of 20 ml of 10% ammonium oxalate followed by addition of 1:1 NH_4OH with stirring until its smell persists. The precipitate is allowed to settle for half an

hour. Then filter using Whatmann No. 41 filter paper and washed with cold H_2O until the washings are free from Chloride and Oxalate (the washing may be tested for Cl- by $AgNO_3$ and for Oxalate by adding a few drops of NH_4OH and 1 ml clear $CaCl_2$ solution. Non-appearance of any precipitate or turbidity shows their absence).

Dissolve the Ca-Oxalate precipitate in H_2SO_4(1:8) (10 ml) opening the filter paper with the precipitate on a watch glass over the beaker and transferring the precipitate by adding drops of H_2SO_4 (1:8) on the filter paper and finally washing with hot water. Add about 150 ml distilled water to dilute the solution. Mixture is to be heated to 60-80°C before titration with standard $KMnO_4$ solution from a burette until the first permanent faint pink colour appears.

Record the titre value and calculate the amount of Ca in gms/litre.

(b) Dissolve A in 20 ml 1.2(N) H_2SO_4 in a 100 ml beaker and add with stirring a solution of B dissolved in minimum volume of distilled water. Filter off undissolved or suspended matter if any. Take the filtrate in a porcelain basin and place the same on a boiling water bath for evaporation. When bottle green crystals of the product start forming on the sides of the porcelain basin, allow the solution to cool down, first in air, up to room temperature and then in an ice bath, when bulk of the double salt crystallizes out. Collect the crystals by filtration

under sucction using a Buchner funnel or a sintered glass funnel,wash with ice-cold (1:1) ethanol-water mixture containing a few drops of dilute(1-2N) H_2SO_4. Drain well and allow the crystals to dry in air.

(Physical Chemistry)

2. Select one experiment from the list of experiments given below by the method of drawing of lots, in presence of examiner, and then perform the experiment that you have chosen. 40

(a) Finding the actual concentration of a supplied solution (approx. N/10) of a dibasic acid by titrating the solution conductometrically against a standard solution of NaOH.

(b) Finding the actual concentration of each acid component in a supplied mixture (total concentration of approx.N/10) of HCl and CH_3COOH acids by titrating the mixture conductometrically against a standard solution of NaOH.

(c) Determination of ionization constant of a weak monoprotic acid by conductometric method.

(d) Estimation of the concentration of an unknown solution of Mohr's salt by potentiometric titration against a standard solution of $K_2Cr_2O_7$, and finding the standard potential of Fe^{3+}/Fe^{2+} redox couple.

(e) Determination of pKa values of a weak dibasic acid by pH-metric method.

(f) Testing the validity of Lambert-Beer's law for $K_2Cr_2O_7$ solution and determining the concentration of the supplied solution of $K_2Cr_2O_7$ by using colorimeter/ spectrophotometer.

(g) Determination of pK_{In} of bromocresol green by using colorimeter.

(h) Determination of the critical solution temperature of phenol-water system and mass percent of phenol at this temperature.

Marks are distributed on the following heats :

Theory of the experiment, Temperature recording (if necessary), Presentation and tabulation of experimental data, calculation, Graph plotting and Results.

(Organic Chemistry)

3. (a) Correlate the marked absorption peaks in the supplied IR-spectrum of a known organic compound to its characteristics structural feature. 1×5

(b) (i) Indicate how many different types of proton are present in the given organic compound and marked them by a, b, c ..... etc. 5×2

(ii) Arrange them according to their chemical shift (δ – ppm).

(iii) Locate each type proton to signal of protons with appropriate chemical shift or range of chemical shift in the supplied H-NMR spectrum of the compound.

(iv) Explain the nature of splitting of signal(s) [If present].

(v) Find out the number of protons in each signal.

4. Place 4 gm of A, 200 ml water, 9 gm of B and supplied liquid 'C' in a 500 ml round bottomed flask equipped with a reflux condenser. Boil the mixture gently for 1 hour. Allow to cool, acidify with conc. HCl (about 40 ml) and add 20% aqueous solution of sodium sulphite hepta hydrate with shaking until it became a colourless solution. Again allow to cool and collect the solid by filtration, washed with cold water. Dry and take the weight. Recrystallize 1/4th of the crude product from hot water. Submit both the crude and recrystallized product separately. 25

Record :

(i) Colour of the crude product ;

(ii) Weight of the crude product ;

(iii) Submission of recrystallized product ;

(iv) Melting point of the recrystallize product.

[Countersign by the examinerFor (ii), (iii) and (iv) is essential]

5. Laboratory Note Book. 5

6. Viva-Voce. 5
