NEW

Part–III 3-Tier

2019

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.
Candidates are required to give their answers in their own words as far as practicable.

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

Inorganic Chemistry (6 Hours)

1. (a) Estimate the total amount of Fe(III) and Cr(VI) quantitatively in the supplied solution marked 'V'. 30

(b) Prepare the following compound as per instruction given below. Report the yield of the dry product and submit the dry product. 10

[Procedure]

1. (a) Estimation of Fe(III) and Cr(VI) in the supplied solutions marked 'V' :

(i) *Preparation of stock solution :*

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

(ii) *Preparation of 250 ml standard (N/10) $K_2Cr_2O_7$ solution :*

Weigh out accurately 0.6125 g $K_2Cr_2O_7$ (A.R. grade) and dissolve it by distilled water in a 250 ml volumetric flask.

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

Pipette out an aliquot of 25 ml of supplied Mohr's salt solution into a 500 ml conical flask and dilute to 150 ml with distilled water. Add 5 ml concentrate H_2SO_4 and 5 ml concentrate H_3PO_4 to the solution. Cool the solution and add 4-5 drops BDS indicator and titrate the resulting solution with standard (N/20) $K_2Cr_2O_7$ solution until the reddish-violet colour appears.

(iv) *Estimation of Fe(III) :*

Pipette out 25 ml of the stock solution into a 500 ml beaker. Dilute the solution to about 100 ml with distilled water. Add 1.0 g solid NH_4Cl,

(Continued)

and heat the solution nearly to boiling. Add dropwise 1:1 aqueous NH_3 with constant stirring till the smell of NH_3 persist. Settle down the precipitation of $Fe(OH)_3$ and then filter through a Whatman No. 41 filter paper. Wash the precipitate with 1% NH_4Cl solution containing a few drops of NH_3 till free from the dichromate (to be tested with a few drops of $AgNO_3$ followed by acidification with acetic acid). Dissolve the precipitate in minimum volume of hot (1:1) HCl and hot distilled water successively. Reprecipitate Fe(III) quantitatively with 1:1 aqueous NH_3 as mentioned earlier and allow to stand for settle down the precipitate. Refilter the precipitate through the same filter paper and wash as before till the washing are colourless. Dissolve the precipitate in 50 ml of hot (1:1) HCl and finally wash with hot distilled H_2O in the same beaker untill the filter paper becomes colourless. Heat the solution to about 70°C–80°C and add small pieces of Al-foil stepwise to reduce Fe(III) to Fe(II), swirl the solution till all the Al-foil gets dissolved giving rise a clear solution. Cool the solution to room temperature and dilute to 150 ml with distilled water. Add 5 ml syrupy H_3PO_4 and 4-5 drops BDS indicator and titrate the solution with the standard (N/20) $K_2Cr_2O_7$ to a radish-violet end point. Record the titre value to calculate the total amount of iron present in the supplied sample.

(v) *Estimation of Cr(VI) :*

Pipette out 25 ml of stock solution into a 500 ml conical flask. Add a measure excess 50 ml of standard Mohr's salt solution ; 50 ml of 4(N) H_2SO_4 and 5 ml of syrupy H_3PO_4 into the flask. Cool the resulting solution under tap if required and dilute to 150 ml using distilled H_2O. Add 4-5 drops of BDS 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 value and calculate the amount of Cr(VI) present in the supplied sample.

[***N.B.:*** 1000 ml 1(N)$K_2Cr_2O_7$ ≡ 55.85 g of Fe^{3+}

1000 ml 1(N)$K_2Cr_2O_7$ ≡ 17.33 g of Cr(VI)]

(b) *Procedure of Inorganic Preparation :*

Dissolve supplied sample A in 20 ml 2(N) H_2SO_4 in a 100 ml beaker and add with stirring a solution of supplied sample 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 suction using a Buchner funnel, or, a sintered glass funnel, wash with ice-cold (1:1) ethanol-water mixture containing a few drops of dilute 2(N) H_2SO_4. Drain well and allow the crystals to dry in air. Report the yield of the dry product.

Physical Chemistry (6 Hours)

2. A. Perform *one experiment* from the list of experiments to be allotted through lottery in presence of the examiners.

Marks are awarded on the following points :

Theory, Temperature recording, Presentation and Tabulation of experimental data, Calculation, Graph plotting, Results. 40

(a) Determine the strength of a supplied solution of a weak dibasic acid [approx 0.1 (N)] by titrating it against a standard solution of NaOH conductometrically.

(b) Determine the strength of HCl and CH_3COOH in the given mixture [total concentration of approx. 0.1(N)] by titrating the mixture against a standard solution of NaOH conductometrically.

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

(d) Determine the strength of the supplied Mohr's Salt solution by titration against a standard $K_2Cr_2O_7$ solution potentiometrically and hence determine the formal reduction potential of Fe^{3+}/ Fe^{2+} redox system.

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

(f) Verify Lambert-Beer's law using $K_2Cr_2O_7$ solutions of different concentrations and determine the concentration of a given $K_2Cr_2O_7$ solution of unknown strength by using colorimeter or spectrophotometer.

(g) Determine of pK_{Dn} of bromocresol green by using colorimeter.

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

B. Viva-Voce 05

C. Laboratory Note Book. 05

Organic Chemistry (6 Hours)

3. A. (a) Correlate the marked absorption peaks in the supplied IR-spectrum of a known organic compound to its characteristic 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 (g) of (A) 20 ml (B) and 10 ml of (C) in a 250 ml round-bottom flask fitted with a long air condenser and heated for 2 hrs in a boiling water bath.

The reaction mixture is then cooled under tap, poured into 100 g crushed ice taken in a 250 ml beaker with stirring. The solid separated is filtered under suction, washed thoroughly with water, recrystallized from rectified or methylated spirit and dried.

Record :

(i) Colour of the crude product ;

(ii) Weight of the crude product ;

(iii) Submission of recrystallized product ;

(iv) Melting point of the recrystallized product.

[Countersigned by the examiner for (ii), (iii) and (iv) is essential]

B. Laboratory Note Book. 5

C. Viva-Voce. 5
