

[TIME: 3 HOURS]

[MARKS: 100]

Please check whether you have got the right question paper

- N.B:
1. All questions are compulsory.
  2. Figures to the right indicate full marks.
  3. Use of log table/non-programmable calculators is allowed.

Q.1 Answer any four of the following:

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- A) Discuss the various grades of laboratory reagents.
- B) Calculate the molarity and molality of 69.8% (w/w) nitric acid solution.  
[Given: molecular weight of nitric acid =63 and density of nitric acid solution =1.42 gm<sup>-3</sup>]
- C) Discuss the importance of quality concepts in industry.
- D) Calculate the percentage composition of each element present in urea.  
[Given: atomic weight of: H=1.008, N= 14.007, O= 15.999 and C= 12.011].
- E) Discuss the different equipments for the sampling of compact solids.
- F) Write a note on: 'Preservation and dissolution of the sample'.

Q.2 Answer any four of the following:

20

- A) What are redox indicators? Discuss the use of diphenylamine as a redox indicator mentioning the role of acid mixture.
- B) 10.0cm<sup>3</sup> of 0.2M Fe (II) solution is titrated with 0.2M KMnO<sub>4</sub> at pH 2 in acidic medium. Calculate the potential:
  - a) at half the equivalence point
  - b) at the equivalence point
  - c) after addition of double the volume of titrant required at the equivalence point.

**Given:**  $E^{\circ}_{\text{Pt}/\text{Fe}^{3+}, \text{Fe}^{2+}} = 0.771\text{V}$  and  $E^{\circ}_{\text{Pt}/\text{MnO}_4^-, \text{Mn}^{2+}} = 1.510\text{V}$ ,  
Comment on the nature of the titration curve.
- C) Discuss the theory of redox indicators.
- D) Calculate the values of pMg when: a) 0.0 cm<sup>3</sup> b) 5.0 cm<sup>3</sup> c) 10.0 cm<sup>3</sup> and d) 11.0 cm<sup>3</sup> of 0.01 M EDTA is added to a 10.0 cm<sup>3</sup> of 0.01 M Mg<sup>2+</sup> buffered to a constant pH of 10.0.  
Draw the nature of titration curve (pMg versus volume of EDTA).  
[Given:  $K_{\text{MY}} = 4.9 \times 10^8$  and  $\alpha_4$  for EDTA at pH of 10.0 = 0.35].
- E) Discuss the use of EDTA as titrant. Write any three advantages of EDTA as a titrant.
- F) What are the different methods to increase selectivity in EDTA titrations? Discuss any two of them in brief.

Q.3 Answer any four of the following:

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- A) Discuss the principle of flame photometric analysis.
- B) With the help of a neat diagram explain an electrothermal atomizer.
- C) Give any three applications and two limitations of AAS.

- D) Derive a mathematical relationship between the intensity of fluorescent radiation and the concentration of the solution.
- E) What is phosphorimetry? What are the factors affecting fluorescence and phosphorescence.
- F) Explain why sample solutions for nephelometry and turbidimetry have to be prepared very carefully. Draw a neat labelled diagram of a nephelometer.

**Q.4 Answer any four of the following:**

**20**

- A) What are the factors affecting solvent extraction? Explain any one.
- B) What is solid phase extraction? Give any three applications.
- C) What are the requirements for high pressure pump used in HPLC? Name any two pumps.
- D) Draw a neat labelled diagram of a typical HPLC unit and explain the function of degasser and precolumn.
- E) Mention the different detectors used in HPTLC and explain any one.
- F) What are the advantages and limitations of HPTLC?

**Q.5A) Answer any five of the following**

**05**

- a) What are certified reference materials?
- b) What is the normality of 0.01M sulphuric acid?
- c) Name any one concentration unit independent of temperature.
- d) What is the condition in which the molarity and formality of the solution will be same?
- e) Mention any one difficulty encountered in the sampling of gases.
- f) What is ambient sampling?
- g) Name the equipment used for sampling of flowing liquids.
- h) Define: 'Bulk ratio' with respect to sample size.

**B) Select the correct option and complete the following statements: (any five)**

**05**

- a) During the direct EDTA titrations, \_\_\_\_\_ is added to metal ion solution to prevent precipitation of hydroxides of metal ion.  
 i) tartarate                      ii) formaldehyde                      iii) sodium cyanide
- b) The transition potential of ferroin indicator is \_\_\_\_\_ V in 1M H<sub>2</sub>SO<sub>4</sub>.  
 i) 0.76                      ii) 1.14                      iii) 0.61
- c) During the titration of Fe<sup>2+</sup> against Ce<sup>4+</sup>, the potential of indicator electrode before the equivalence point depends on the ratio of \_\_\_\_\_.  
 i)  $\frac{[Fe^{2+}]}{[Fe^{3+}]}$                       ii)  $\frac{[Ce^{3+}]}{[Ce^{4+}]}$                       iii)  $\frac{[Fe^{2+}]}{[Ce^{4+}]}$
- d) EDTA is standardized using standard solution of \_\_\_\_\_.  
 i) ZnSO<sub>4</sub>                      ii) NaOH                      iii) HCl
- e) Of the following, \_\_\_\_\_ is estimated by indirect EDTA titrations only.  
 i) PO<sub>4</sub><sup>3-</sup>                      ii) Cu<sup>2+</sup>                      iii) Zn<sup>2+</sup>

- f) Eriochrome Black T indicator exhibits \_\_\_\_\_ colour between pH of 7 to 11.  
i) red                      ii) blue                      iii) orange
- g) Ferroin indicator is a complex of with \_\_\_\_\_ with Fe(II).  
i) 1,10-phenanthroline      ii) diphenylamine      iii) diphenylbenzidine

C) **State whether true or false: (any five)**

05

- a) Nitrogen is used as an inert gas in hollow cathode lamp.  
b) Flame photometry gives the information of the molecular condition of the sample.  
c) Flame photometry cannot be used for analysis of non-radiating elements.  
d) Turbidimetry is used to measure the amount of growth of a test bacteria in a liquid nutrient medium.  
e) Fluorescence is delayed phosphorescence.  
f) Phosphorescence of a solution is generally observed at liquid nitrogen temperature.  
g) For molecular weight determination by turbidimetry, a plot of turbidance versus concentration is plotted.  
h) In nephelometric analysis, highly monochromatic radiation is not necessary.

D) **Select the correct option and complete the following statements: (any five)**

05

- a) When the separation factor for the two solutes are very close, \_\_\_\_\_ extraction method is used.  
(batch , continuous, counter current)
- b) Partition coefficient is equal to \_\_\_\_\_ if the molecular state of the solute remains unchanged.  
(distribution ratio, separation factor, equilibrium constant)
- c) If the  $pH_{1/2}$  values of two metals are \_\_\_\_\_, excellent separation is achieved by controlling the pH.  
(very close, similar, very far)
- d) Due to different \_\_\_\_\_ of the solutes in between the two liquid phases, separation of the solutes occur in HPLC.  
(solubilities, adsorption, absorption)
- e) In HPLC, the \_\_\_\_\_ at which the peak occurs on the chromatogram is characteristic of the analyte.  
(concentration, retention time, peak area)
- f) \_\_\_\_\_ detector used in HPLC is highly temperature sensitive.  
(UV, Refractive index, Fluorescence)
- g) The \_\_\_\_\_ method of sample injection in HPLC is reliable with respect to reproducibility.  
(manual, stop flow, sample loop )

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