

(Time: 3 hours)

Total Marks: 100

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

**Q.1** **Attempt any four of the following.** **20**

- A) Discuss various grades of laboratory reagents.  
 B) Calculate mass percent composition of each element in  $\text{CH}_3\text{CH}_2\text{OH}$ , ethanol molecule.  
 (Given: atomic mass of C = 12, H = 1, O = 16)  
 C) Calculate the number of grams of pure  $\text{Na}_2\text{CO}_3$  required to prepare  $250 \text{ cm}^3$  of 0.1 N solution. This  $\text{Na}_2\text{CO}_3$  is to be titrated with HCl according to equation  $\text{CO}_3^{2-} + 2 \text{H}^+ \rightarrow \text{H}_2\text{CO}_3$   
 (Given: atomic mass of Na = 23, C = 12, O = 16)  
 D) Describe the sampling of homogeneous and heterogeneous liquid.  
 E) Name different methods of reduction of sample size in sampling of solid. Explain any two of them.  
 F) Explain the terms quality and quality control.

**Q.2** **Attempt any four of the following.** **20**

- A) What are the advantages and limitations of EDTA as titrant?  
 B) What are redox indicators? Explain the use of iron(II) orthophenanthroline indicator.  
 C) Calculate the potential of the system in the titration of  $10 \text{ cm}^3$  of 0.2M Fe(II) against 0.2M Ce(IV) solution when:  
 i)  $12 \text{ cm}^3$  Ce(IV) solution added.  
 ii)  $15 \text{ cm}^3$  Ce(IV) solution added.  
 ( $E^0_{\text{Pt}|\text{Fe}^{2+},\text{Fe}^{3+}} = +0.771 \text{ V}$ ,  $E^0_{\text{Pt}|\text{Ce}^{3+},\text{Ce}^{4+}} = +1.440 \text{ V}$ )  
 D) How is the selectivity is enhanced in the complexometric titrations by the following:  
 i) kinetic masking ii) use of masking and demasking agents.  
 E) What are metal-ion indicators? What are their requirements?  
 F) Derive an equation for potential of the system at equivalence point in the titration of Fe(II) against  $\text{MnO}_4^-$ .

**Q.3.** **Attempt any four of the following.** **20**

- A) Discuss the principle of Atomic Absorption Spectroscopy. Explain the role of rotating chopper in AAS.  
 B) Discuss the basic principles of FES.  
 C) Explain the applications of fluorescence spectroscopy.

- D) Explain the principles underlying the emission of fluorescent light. How does fluorescence differ from phosphorescence?
- E) Draw a schematic diagram of turbidimeter and explain turbidimetric titrations using turbidimetric titration curve.
- F) What are the important factors affecting the scattering of radiation? Explain any two.

**Q.4 Attempt any four of the following. 20**

- A) Explain any two factors affecting solvent extraction.
- B) Discuss the different steps involved in process of solid phase extraction.
- C) Explain the terms used in HPLC i) isocratic elution ii) gradient elution. Give any three applications of HPLC.
- D) With a neat and labelled diagram explain the working of HPLC.
- E) Give applications and limitations of HPTLC.
- F) Explain double beam densitometer used in HPTLC. Give any two advantages of HPTLC.

**Q.5 A) Select the correct option and complete the following statements: (any five) 05**

- a) \_\_\_\_\_ material can be used for verification of method validation parameters.  
i) Reference material ii) Certified reference material iii) LR grade
- b) The next step after quality control is \_\_\_\_\_.  
i) quality management ii) quality assurance iii) quality development
- c) The sum of mole fraction of solute and solvent is \_\_\_\_\_.  
i) 1 ii) 1.5 iii) 2.5
- d) \_\_\_\_\_ is a method of expressing concentration in a solution on weight basis.  
i) Normality ii) Molality iii) Molarity
- e) Flushing method is used for sampling of \_\_\_\_\_.  
i) Solid ii) liquid iii) gases
- f) \_\_\_\_\_ is used for sampling of compact solid.  
i) Auger sampler ii) Multiple tube sampler iii) Split tube thief
- g) The ratio of weight of sample to total weight of bulk is \_\_\_\_\_.  
i) Bulk size ii) Size:weight ratio iii) Bulk ratio
- h) \_\_\_\_\_ is used for sampling of flowing liquid.  
i) Concentric tube thief ii) Geo-sampler iii) multiple tube sampler

**Q.5 B) State whether true or false: (any five) 05**

- a) In the titration of iron(II) with cerium(IV); iron(II) undergoes reduction.
- b) Potassium permanganate acts as self indicator in redox titrations.
- c) Nernst's distribution law is applicable in redox titrations.
- d) pCa is defined as  $\log_{10}[\text{Ca}^{+2}]$
- e) Diphenyl amine is the first indicator to be used in redox titrations.

- f) Dissociation of EDTA is not affected by pH.
- g) Murexide is obtained from purpuric acid.

**Q.5 C) Fill in the blanks with correct alternatives given in the bracket: 05 (any five)**

**(acetylene, phosphorescence, turbidity coefficient, turbidance, toxic metals, mist, low, right)**

- a) AAS is used to detect \_\_\_\_\_ like Cu, Ni, Zn and Hg in food products.
- b) The fuel used in a premix burner in FES is \_\_\_\_\_.
- c) The nebulizer converts the sample solution into a \_\_\_\_\_.
- d) Delayed re-emission of absorbed radiation is called \_\_\_\_\_.
- e) Phosphorimetric experiments are normally carried out at \_\_\_\_\_ temperature.
- f) In the expression w.r.t. turbidimetry,  $S=Ktc$ , 'K' stands for \_\_\_\_\_.
- g) In nephelometry, the detector is usually, but not necessarily, placed at \_\_\_\_\_ angle to the incident radiation.
- h) A turbidimeter measures \_\_\_\_\_ as a function of concentration of suspensions.

**Q.5 D) Match the columns: (any five) 05**

**Column A**

**Column B**

- |   |   |
|---|---|
| a) TBP solvent  | (i) Ion-pair formation                    |
| b) Multistage separation                                  | (ii) Easily extracted in organic solvent  |
| c) Hydrophobic functional groups chemically bonded silica | (iii) Analytical column                   |
| d) Neutral chelate  | (iv) HPTLC                                |
| e) Complexes form clusters                                | (v) Solid phase extraction                |
| f) HPLC   | (vi) $pH_{1/2}$                           |
| g) Densitometer   | (vii) Easily extracted in aqueous solvent |
|   | (viii) Countercurrent extraction          |
|   | (ix) Extraction of uranyl nitrate         |

\*\*\*\*\*