

[Time: 2 $\frac{1}{2}$ Hours]

[Marks:75]

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 calculator is allowed.

Q 1. Answer any three of the following.**15**

- A) Discuss the interference of dissolved oxygen, with reaction involved in polarographic analysis, explaining its removal.
- B) In polarographic determination, a series of standard solutions and an unknown solution containing cadmium ions were prepared and the wave heights were determined. The results were as follows.

Concentration (mM)	0.40	0.80	1.20	unknown
Wave height (mm)	28.0	55.9	83.9	42.0

Calculate the concentration of the unknown solution.

- C) Mention any five advantages of DME over other micro electrodes.
- D) In polarographic estimation of Cu^{2+} ions, the following results were obtained.
 $i_d = 5.65 \mu\text{A}$, $D = 6.85 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$, $m = 3.45 \text{ mgs}^{-1}$ and $t = 2.9\text{s}$. Calculate the concentration of Cu^{2+} ions in the sample solution.
- E) Give any five applications of amperometric titrations.
- F) Explain the nature of amperometric titration curve when titrand and titrant both are reducible, using suitable example.

Q 2 Attempt any three of the following.**15**

- A) What is the role of detector in GC? Explain flame ionization detector with help of a labelled diagram.
- B) Explain the following with respect to GC:
 (a) HETP, and (b) Eddy diffusion
- C) Draw a neat labelled diagram of HPLC and explain the isocratic and gradient elution.
- D) Give any five applications of HPLC.
- E) What is ion-exchange capacity of ion exchange resin? How is it determined for a cation exchange resin?
- F) What are the requirements of an ideal ion-exchange resin?

Q 3 Answer any three of the following**15**

- A) What is normal error curve? Describe its salient features.
- B) Five replicate measurements for the determination of iron in a sample of iron gave the following results.

15.61, 15.52, 15.63, 15.58, and 15.64% iron.

Calculate the 95% confidence interval for mean if no additional information about the precision of the method is known.

Given : $t = 2.78$, for 95% confidence limit

- C) The following table gives dependence of y on x

x	0	1	2	3	4	5
y	0.0	2.2	4.5	7.1	9.6	11.6

Assuming a linear relationship between the variables, obtain an equation of the type $y=mx$, using the method of averages.

- D) What are different methods used to increase the selectivity of EDTA in EDTA titration? Discuss any two of these in brief.
- E) Explain the following w.r.t. EDTA titrations.
i) Direct titrations, and
ii) Back titrations
- F) Discuss the theory of redox indicators, explaining the basis of selection of an indicator in redox titrations.

Q. 4 Answer any three of the following:**15**

- A) Explain the term quality control and quality assurance.
- B) Explain the term: i) GLP, and
ii) ISO.
- C) Discuss the basic principle of mass spectrometry.
- D) Draw the schematic diagram of the TGA set up. Give any three requirements of a good thermobalance.
- E) What is a thermogram? Discuss the nature of thermogravimetric curve obtained in the thermal decomposition of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.
- F) What are the applications of NAA?

Q. 5 A) Fill in the blanks**04**

- a) Diffusion current = Limiting current - _____.
- b) In DME when the mercury drop gets detached, the current becomes _____.
- c) In polarography, the electrode of large surface area which retains its own potential is called _____ electrode.
- d) Triton X-100 is used for suppressing the polarographic _____.

OR**B) State true or false.****04**

- p) The concentration of supporting electrolyte should be at least 100 times less than the electroactive species.
- q) Diffusion current depends on temperature.
- r) In polarography, conditions are so maintained that the movement of ions to DME is controlled by concentration gradient as well as potential gradient.
- s) Residual current in polarography is regarded as non-faradic current.

Q.5 B) Fill in the blanks**04**

- a) As the number of theoretical plates in the GC column increases, the efficiency of the column _____.
- b) The measure of separation between adjacent peaks in a GC chromatogram is called _____.
- c) In HPLC, precolumn prevents the stripping of the _____ phase from the analytical column.
- d) The equilibrium constant, obtained by applying Law of mass action to a reaction involving ion exchange equilibrium is called as _____.

OR**B) State true or false.****04**

- p) The carrier gas used in GC separations should not react with the sample.
- q) GC is used only for volatile and thermally stable compounds.
- r) Trace metal ions in sea water can be concentrated by ion-exchange chromatographic technique.
- s) Higher the concentration of ions in the solution lower is the exchange in ion exchange chromatography.

Q. 5 C Fill in the blanks.**04**

- a) EDTA contains _____ donor atoms.
- b) Metal indicator complex should be slightly _____ stable than metal EDTA complex in EDTA titrations.
- c) In redox titration of Fe (II) Vs. $\text{Cr}_2\text{O}_7^{2-}$, diphenylamine is first irreversibly oxidized to _____.
- d) A redox titration curve is a plot of _____ versus volume of titrant.

OR**C State true or false.****04**

- p) Redox titration involve transfer of electrons between reactant and titrant.
- q) 1,10 phenanthroline iron (II) sulphate is commonly known as ferroin indicator.
- r) Concentration of SO_4^{2-} can be determined with the help of EDTA.
- s) EDTA titrations are always carried out in basic medium.

Q. 5 D Fill in the blanks**03**

- a) In a mass spectrometer, a _____ separates ions according to their masses.
- b) The decomposition temperature in TG increases with _____ in particle size.
- c) In neutron activation analysis, stable nucleides are converted into _____ isotopes.

OR**D State true or false****03**

- p) A high vacuum is required to be maintained in mass spectrometer.
- q) Multi-elemental analysis is possible by NAA.
- r) In thermogravimetric technique, it is necessary to avoid fast heating rate to detect intermediate compounds.
