T.-V.BSC

Subject: Chemistry P IV
Analytical Chemistry

2016-17

Sem: VI

QP Code : 77243

(9)

(3 Hours) **[Total Marks : 75**

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.

1. Answer any **three** of the following :– 15

A. What is the basis of qualitative polarographic analysis? Explain its significance with the help of neat labelled polarographic wave giving its equation.

B. 5.0 mM solution of Cd(II) ion gave the diffusion current of 40 μA at constant temperature. Find the diffusion coefficient of Cd(II) ion.
Given : rate of flow of mercury = 3.2 mg s^{-1}, drop time = 2.2 s.

C. Explain the following w.r.t. polarographic analysis.
(i) Function of supporting electrolyte
(ii) Need to pass nitrogen gas through experimental solution.

D. Diffusion current of lead in an unknown solution was 5.6 μA. 1.0 cm^3 of 1×10^{-3} M lead solution was added to 10.0 cm^3 of the unknown solution and diffusion current increased to 12.2 μA. Calculate the concentration of lead in the unknown solution.

E. What are amperometric titrations? Explain the nature of amperometric titration curve when titrand alone is reducible, giving suitable example.

F. Discuss the advantages and limitations of amperometric titrations.

2. Answer any **three** of the following :– 15

A. What is the role of detector in gas chromatography? Describe flame ionisation detector with the help of a labelled diagram.

B. Explain HETP w.r.t. GC. Discuss quantitative analysis in GC.

C. Distinguish between GLC and HPLC.

D. Explain isocratic and gradient elution in HPLC. Discuss the refractive index detector used in HPLC mentioning its advantage.

E. What are ion-exchange resins? What are the requirements of an ideal ion-exchange resin?

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F. What is ion-exchange chromatography? Discuss the applications of ion-exchange chromatography w.r.t.
(i) seperation of amino acids
(ii) deionisation of water

15

3. Answer any **three** of the following :–

A. Explain Normal error curve giving its equation. Give any three salient features of this curve.

B. The following table gives the dependence of y on x.

x	0.0	1.0	2.0	3.0	4.0	5.0
y	0.0	2.4	4.7	7.3	9.8	11.8

Assuming a linear relationship between the variables, make a least square analysis of the data to derive an equation of the straight line.

C. What are metal ion indicators? Discuss the theory of metal ion indicators.

D. Two independent methods were used to analyse the sample for its magnesium content. The results gave percentage of magnesium as follows :

Method I	5.94	6.01	5.98
Method II	5.65	5.70	5.61

The combined standard deviation was 0.03%. Determine whether two methods differ numerically or statistically also.
Given $t = 2.78$, at 95% probability level.

E. Name the different types of EDTA titrations. Explain any two of these types in brief.

F. What are redox indicators? Discuss the use of ferroin indicator in the redox titration of Fe(II) versus Ce(IV)

4. Attempt any **three** of the following :– 15

A. What do you mean by ISO? Describe ISO 9000 series as total quality system.

B. Explain 'Quality Assurance'. How does it differ from quality control?

C. Draw a schematic diagram of a mass spectrometer. Explain the role of ion source and ion separator.

D. Explain the various factors affecting a thermogravimetric curve.

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E. Discuss the important applications of TGA.

F. Explain the principle underlying neutron activation analysis. 4

(A) Fill in the blanks :–

(a) The quantitative relation between diffusion current and the concentration of electroreducible species in given by _____ equation.

(b) Limiting current - Residual current = _____.

(c) The polarisable electrode used in polarography is _____.

(d) The unit of diffusion coefficient in quantitative polarographic expression is _____.

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OR

(A) State true or false :–

(p) Migration current is eliminated during polarographic analysis with the help of indifferent electrolyte.

(q) Triton X-100 is used as maxima suppresor in polarographic analysis.

(r) The magnitude of half wave potential is independent of supporting electrolyte used.

(s) Since DME surface is continuously renewed, a series of reducible species can be estimated in the given solution. 4

(B) Fill in the blanks :–

(a) In GC, the band broadening factor caused by random motion of solute molecules through the column is called _____ diffusion.

(b) In GLC, the seperation is based on the _____ of the solute between the gaseous mobile phase and the liquid stationary phase.

(c) In HPLC, the purpose of _____ is to remove impurities from the solvent in order to prevent contamination of analytical column.

(d) Capacity of ion-exchange resin is measured in terms of _____ of resin.

OR

4

(B) State true or false :–

(p) The temperature of GC column is adjusted slightly above the boiling point of components of the sample.

(q) In GC, greater the affinity of the solute for the stationary phase, greater will be retention time of the solute in the column.

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(r) The exchange capacity of a weakly acidic cation exchanger increases with decrease in pH.

(s) Ion-exchange chromatography is useful for the seperation of essential oils. 4

(C) Fill in the blanks :–

(a) Due to low solubility of EDTA in water, its _____ dihydrate salt is commonly used for titration.

(b) An ideal redox indicator should change colour, when titrant causes a shift of potential of about _____ V.

(c) In redox titration of Fe(II) v/s $K_2Cr_2O_7$, diphenylamine is first irreversibly oxidised to _____.

(d) A solution containing Bi^{3+} and Pb^{2+} ions is titrated for _____ ion at pH 2 using xylenol orange indicator. 4

OR

(C) State true or false :–

(p) EDTA reacts with Zn^{2+} ions in the ratio 1:1.

(q) Formaldehyde is used as a masking agent in EDTA titration.

(r) In the titration of Fe(II) v/s $K_2Cr_2O_7$, diphenylamine indicator would show a colour change much before the equidence point, in the absence of H_3PO_4.

(s) To see the colour of oxidised form of redox indicator,

$$\frac{[In(ox)]}{[In(red)]} \geq 10.$$ 3

(D) Fill in the blanks :–

(a) The motto of quality control should be no _____ without analysis.

(b) The pyrolysis curve is a plot of change in _____ of the sample against temperature.

(c) NAA cannot be used for the analysis of elements whose radioisotopes have a very short _____ period.

OR 3

(D) State true or false :–

(p) The laboratory should be provided with air conditioning facility is one of the requirements for GLP.

(q) Differential thermal analysis is a type of thermoanalytical method.

(r) NAA cannot be used for the analysis of cadmium.
