

- N. B. : (1) All questions are compulsory.
 (2) Figures to the right indicate full marks.
 (3) Use of logarithmic table/non programmable calculator is allowed.
 (4) Atomic weight: H=1, C=12, N=14, O=16, Na=23, Al=27, S=32,
 Cl=35.5, K=39, Ca=40, Ag=108, Ba=137, Pb=207,
 Mg= 24.3, F=19 Ni = 58.7, Cr = 52, Zn = 65.4

1. (a) Attempt any two of the following:

- (i) What do you mean by calibration of instrument? Describe briefly the use of internal standard method for calibration. What difficulties are encountered in this method? 4
 (ii) What is 'Quality Manual'? List the activities that should be included in Quality Manual. 4
 (iii) Define 'Sensor' with respect to analytical device. Discuss quartz crystal microbalance in detail. 4
 (iv) Define the terms 'Quality Audit' and 'Quality Review'. Discuss the different types of quality audit. 4

(b) Attempt any one of the following:

- (i) Define the term 'Quality'. Discuss the role of quality manager in analytical laboratory. 4
 (ii) A least square analysis of calibration data for the determination of lead based upon its atomic absorption spectrum yielded the equation $S = 1.43 C_{Pb} + 0.543$, where C_{Pb} is the concentration of lead in parts per million and S is a measure of relative intensity of the lead emission line. 4

The following replicate data were then obtained;

Conc. (ppm) of Pb	No. of replications	Mean value of S	Standard deviation
10.0	10	15.12	0.15
1.00	10	5.12	0.015
0.000	25	0.029	0.008

Calculate

- (1) the calibration sensitivity
- (2) the analytical sensitivity at 1.0 ppm and 10 ppm of Pb
- (3) the detection limit.

2. (a) Attempt any two of the following:

(i) (1) How much of potassium nitrate must be weighed to prepare a solution containing 7 mg of Na^+ per liter? 2

(2) A solution contains 0.15 kg each of water and ethanol. Calculate mole fraction of each component. 2

(ii) Calculate the number of millimoles of chloride ions required to react with sufficient amount of AgNO_3 to produce 4.5 g of silver chloride. What will be the mass of CaCl_2 required to generate the required amount of chloride ions? 4

(iii) The solubility of magnesium fluoride in 250 cm^3 of water is 0.01869 gm. Find its solubility product. 4

(iv) A solution of 500 cm^3 of $1 \times 10^{-3} \text{ M NiCl}_2$ (aq) is titrated with $1 \times 10^{-3} \text{ M EDTA}$ in a solution of 0.1 M ammonia at pH 11.0. Calculate pNi when 25.0 cm^3 of titrant solution is added? ($\alpha_{\text{Ni}^{2+}} = 1.34 \times 10^{-4}$ at 1.0 M ammonia) 4

(b) Attempt any one of the following:

(i) 20 cm^3 of 0.2 M silver nitrate solution is mixed with 50 cm^3 of 0.05 M K_2CrO_4 solution. Calculate the concentration of K^+ , Ag^+ , NO_3^- and CrO_4^{2-} at equilibrium. [Given K_{sp} of $\text{Ag}_2\text{CrO}_4 = 1.12 \times 10^{-12}$] 4

(ii) (1) What is the molarity of 5 ppm solution of Pb^{2+} ion? 2

(2) Calculate mass of anhydrous HCl in 5 cm^3 of HCl (density = 1.19 g/cm^3) containing 37.23% HCl by weight. 2

3. (a) Attempt any two of the following:

(i) With the help of neat labeled diagram describe the construction and working of fluorimetric detector used in HPTLC. 4

(ii) Describe with suitable examples, extraction by solvation. What are limitations of liquid-liquid extraction? 4

(iii) What is extraction by chelate formation? Discuss the factors affecting chelate formation. 4

[TURN OVER

(iv) Derive mathematical expression to relate distribution ratio (D) to pH of aqueous solution in solvent extraction involving chelation. 4

(b) Attempt any one of the following:

(i) What is difference between liquid-liquid extraction and solid phase extraction? Describe the technique of solid phase extraction. 4

(ii) 150 cm³ of an aqueous solution containing 95 mg of solute was extracted once with 10 cm³ of chloroform and 70% of solute was transferred to chloroform. Calculate the amount of solute that will remain behind in aqueous phase after 3 extractions, if 10 cm³ of chloroform was used for each extractions.

4. (a) Attempt any two of the following:

(i) With the help of neat labeled diagram, describe construction and working of pneumatic pump used in HPLC. 4

(ii) Give a comparative account of WCOT, SCOT and FSOT columns. 4

(iii) How do gas-liquid and gas solid chromatography differ? What kind of mixtures are separated by gas solid chromatography? 4

(iv) What are the characteristics of ideal carrier gas used in gas chromatography? Give an account of any one carrier gas used in gas chromatography. 4

(b) Attempt any one of the following:

(i) What are the factors that are to be considered for the selection of liquid stationary phase in GLC? 4

(ii) Calculate the resolution of the peaks of the two components A and B in the chromatograms with peak widths of 0.57 minutes and 0.64 minutes respectively, with the corresponding retention times of 10.42 minutes and 11.54 minutes respectively. Is the resolution adequate for the analysis? What should be retention time of B to achieve the resolution of 1.5? 4

[TURN OVER

5. Attempt any four of the following:

12

- (a) How to choose an analytical method for chemical analysis?
- (b) Discuss Quality assurance and quality control with respect to quality management system in analytical laboratory.
- (c) What volume of HCl of concentration 2 mol/dm^3 would have to be added to 25 cm^3 of 0.5 mol/dm^3 sodium carbonate solution to produce a neutral solution?
- (d) Calculate number of molecules present in 5 dm^3 of N_2 gas at S.T.P.
- (e) Explain the principle of electrochromatography.
- (f) What are factors affecting extraction by chelation? Describe any one in detail.
- (g) Why is temperature programming used in gas chromatography?
- (h) On the basis of van Deemter equation, explain the effect of mass transfer between the phases on band broadening.

12/07/2016 9:55:00 AM MUPD16088 DES088 12/07/2016 9:55:00 AM MUPD16088 DES088 12/07/2016 9:55:00 AM