

- 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) At. Wts : H=1, C=12, N=14, O=16, F = 19, Na=23, Mg = 24.3, Al=27, S=32, Cl=35.5, K=39, Ca=40, Ag=108, Ba=137, Pb=207.

(a) Attempt any two of the following :—

- (i) With respect to quantitative performance criteria of an analytical instrument, explain the terms, 'Calibration sensitivity' and 'Analytical sensitivity'. What are the advantages of analytical sensitivity? 4
- (ii) Discuss the cost and benefits of introducing quality system in laboratory. 4
- (iii) What is calibration of instrument? Describe briefly the use of internal standard method for calibration. What difficulties are encountered in this method? 4
- (iv) What is Quality audit? How is it carried out? 4

(b) Attempt any one of the following :—

- (i) Discuss the responsibilities of staff at all levels towards laboratory quality. 4
- (ii) Calculate the figures of merit for precision, when the following results were obtained in replicate measurement of Fe in a steel sample. Concentration of Fe in ppm, 0.532, 0.533, 0.535, 0.534, 0.535. 4

(a) Attempt any two of the following :—

- (i) (1) How many grams of NaCl should be weighed to prepare 1 liter of a 20 ppm solution of Na⁺? 2
- (2) How many equivalents and milliequivalent of HNO₃ are present in 75cm³ of 0.2 N solution? 2
- (ii) 50.0 kg of N₂(g) and 10.0 kg of H₂(g) are mixed to produce NH₃(g). Identify the limiting reagent in the production of NH₃ in this situation. Calculate the amount of ammonia formed in the process. 4
- (iii) 1.1 gm of an impure sample of soda ash (impure Na₂CO₃) when titrated with 0.5 N H₂SO₄, required 35.0 cm³ of the acid for complete neutralization. What is the percentage of Na₂CO₃ in the sample, if no other active component was present in the sample? 4
- (iv) The solubility of magnesium fluoride in 250cm³ of water is 0.01869gm. Calculate its solubility product. 4

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(b) Attempt any one of the following :—

- (i) Determine the molarity of each ion in a saturated solution of $Mg_3(PO_4)_2$. 4
 Given: K_{sp} for $Mg_3(PO_4)_2 = 1.0 \times 10^{-13}$
- (ii) Calculate the mass of sodium acetate that should be added to 0.5 dm^3 of an aqueous solution containing 0.02 mol of acetic acid to obtain a buffer solution of $\text{pH} = 4.5$. (Given : K_a for acetic acid = 1.8×10^{-5}) 4

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

- (i) Derive mathematical expression to relate distribution ratio (D) to pH of aqueous solution in solvent extraction involving chelation. 4
- (ii) Discuss any two factors affecting extraction by ion-pair formation. 4
- (iii) What are the advantages and limitations of solvent extraction? What are the advantages of solid phase extraction over solvent extraction? 4
- (iv) Describe the technique of countercurrent extraction. 4

(b) Attempt any one of the following :—

- (i) 200 cm^3 of an aqueous solution containing 120 mg of solute, when extracted once with 50 cm^3 of an organic solvent, 84 mg of the solute was transferred to the organic solvent. Calculate the percent extraction after three extractions, by using same volume of organic solvent for each extraction. 4
- (ii) With the help of neat labeled diagram describe the construction and working of double beam densitometer used in HPTLC. 4

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

- (i) Give an account of different columns used in GLC. 4
- (ii) With the help of neat labeled diagram, describe construction and working of pneumatic pump used in HPLC. What are its advantages and limitations? 4
- (iii) What are the characteristics of ideal carrier gas used in gas chromatography? Why hydrogen or helium is used as carrier gas in thermal conductivity detector? 4
- (iv) With the help of neat labeled diagram, describe construction and working of electrochemical detector used in HPLC. 4

(b) Attempt any one of the following :—

(i) The following data was obtained on a chromatographic column having a length of 25.0 cm. 4

(1) The unretained species had a retention time of 1.10 min.

(2) Retention times of the component X was 8.20 min.

(3) The V_M and V_s of the column were 12.2 cm³ and 42.4 cm³ respectively.

Calculate capacity factor and distribution constant of X.

(ii) What is the difference between (1) normal phase chromatography and reverse phase chromatography (2) isocratic elution and gradient elution? 4

3. Attempt any four of the following :—

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(a) Explain the following terms with respect to analytical instrument

(i) Detector (ii) Transducer (iii) Sensor

(b) Discuss the types of quality standards for quality systems in laboratories.

(c) The values of K_{sp} of two sparingly soluble salts $Ni(OH)_2$ and $AgCN$ are 2.0×10^{-15} and 6×10^{-17} respectively. Which salt is more soluble? Explain.

(d) The density of 37.23%(w/w) solution of HCl is 1.19 g/cm³. Calculate the mass of HCl in 50.0 cm³ of the above solution.

(e) Explain the principle of ion exchange chromatography.

(f) Distinguish between TLC and HPTLC.

(g) On the basis of van Deemter equation, explain the effect of longitudinal diffusion on band broadening.

(h) Why is temperature programming used in gas chromatography?

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