

संचालित प्रश्नपत्रिका तपासून घ्याव्यात  
नंतर तक्रार घालणार नाही.  
परत घेतलेली तपासून घेतल्या जातील.

(2½ Hours)

[ Total Marks : 60

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, Na = 23, Al = 27, S = 32, Cl = 35.5,

K = 39, Ca = 40, Cr = 52, Fe = 56, Ba = 137, Pb = 207

(a) Attempt any two of the following:

- (i) With reference to performance characteristics of instrument explain the following terms: 4  
1) Bias 2) Detection limit 3) Sensitivity
- (ii) Define the term 'quality'. Discuss the role of quality manager in analytical laboratory. 4
- (iii) What do you understand by calibration of an instrument? Describe briefly, the use of internal standard method for calibration. What difficulties are encountered in this method? 4
- (iv) What is an internal audit? How is it carried out? Who is responsible for an internal audit? 4

(b) Attempt any one of the following:

- (i) With respect to quality, explain 'Quality audit' and 'Quality review'. 4
- (ii) 25.0 cm<sup>3</sup> sample containing Pb<sup>+2</sup> gave an instrumental signal of 18.4 units (corrected for a blank). When exactly 0.5 cm<sup>3</sup> of 0.035 M Pb(NO<sub>3</sub>)<sub>2</sub> was added to the solution, the signal increased to 26.9 units. Calculate the molar concentration of Pb<sup>+2</sup> assuming that the signal was directly proportional to the analyte concentration. 4

2 (a) Attempt any two of the following:

- i) 1) Calculate the amount of chromium in milligrams present in 400 cm<sup>3</sup> of 80 ppm K<sub>2</sub>CrO<sub>4</sub> solution. 2
- 2) Commercially available HCl contains 36% w/w of HCl. Calculate its mole fraction. 2
- ii) In an experiment a mixture of 0.28 mole of methane was burnt in 1.05 mole of oxygen in a sealed vessel. Find the limiting reagent. Calculate theoretical yield of water in grams. 4

- iii) A sample of pure  $\text{CaCO}_3$  (M.W. 100.09) weighing 0.425 gm was dissolved in 1:1 hydrochloric acid, and the solution was diluted to  $250 \text{ cm}^3$ .  $25 \text{ cm}^3$  of the above solution was titrated with EDTA solution using EBT indicator. A volume of  $20.4 \text{ cm}^3$  was required to reach end point. Calculate molarity of the EDTA solution. 4
- iv) Determine molarity of each ion in a saturated solution of  $\text{Mg}_3(\text{PO}_4)_2$ . (Given  $K_{sp}$  for  $\text{Mg}_3(\text{PO}_4)_2 = 1.0 \times 10^{-13}$ ). 4
- b) Attempt any one of the following:
- i) 15 millimole of solid NaOH was added to  $75 \text{ cm}^3$  of 0.2 M formic acid. Calculate pH of the resultant solution ( $\text{pK}_a$  for formic acid = 3.77). 4
- ii) 1.5 mol of  $\text{H}_2$  was mixed with 1.0 mol  $\text{I}_2$  in 100.0 liter container and allowed to react at  $448^\circ\text{C}$ . The equilibrium constant for the reaction  $\text{H}_{2(g)} + \text{I}_{2(g)} \rightarrow 2\text{HI}_{(g)}$  is 50.0 at  $448^\circ\text{C}$ . How many moles of HI will be formed at equilibrium? How many moles of  $\text{H}_2$  and  $\text{I}_2$  will remain unreacted? 4
- 3 a) Attempt any two of the following:
- i) What are the advantages and limitations of solvent extraction? What are the advantages of solid phase extraction over solvent extraction? 4
- ii) Derive mathematical expression to relate distribution ratio (D) to pH of aqueous solution in solvent extraction involving chelation. 4
- iii) Explain with suitable example use of crown ethers in solvent extraction. 4
- iv) Discuss any two factors affecting extraction by ion pair formation. 4
- b) Attempt any one of the following:
- i) With the help of neat labeled diagram describe the construction and working of fluorimetric densitometer used in HPTLC. 4
- ii)  $200 \text{ cm}^3$  of an aqueous solution containing 200 mg of solute, when extracted once with  $25 \text{ cm}^3$  of ether, 150 mg of solute was transferred to ether. Calculate percentage extraction after three extractions if same volume of ether was used for each extraction. 4

- 4 (a) Attempt any two of the following:
- With the help of neat labeled diagram, describe construction and working of pneumatic pump used in HPLC. 4
  - Why high pressure pumps are necessary in HPLC? What are the functions of guard column in HPLC? 4
  - Give an account of different columns used in GLC. 4
  - 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:
- Describe the working of a detector which is selective for the determination of pesticide containing phosphorous, by gas chromatography. 4
  - Calculate the resolution of the peaks of the two components 'A' and 'B' in the chromatograms with peak widths of 0.64 and 0.75 minutes respectively, with the corresponding retention times of 11.52 and 12.26 minutes respectively. Is the resolution adequate for the analysis? What should be retention time of 'B' to achieve the resolution of 1.5? 4
5. Attempt any four of the following: 12
- How to choose an analytical method for chemical analysis?
  - Discuss the benefits of introducing quality system in laboratory.
  - How much 0.6M Oxalic acid is required to neutralize 100 cm<sup>3</sup> of 0.325M aluminum hydroxide?
  - What is the solubility product of silver sulphate (Ag<sub>2</sub>SO<sub>4</sub>). (Formula weight = 312), if the solubility of the salt is 5.7x10<sup>-3</sup>g per cm<sup>3</sup>?
  - Explain the principle of ion exchange chromatography.
  - Discuss in brief :  
(1) sample preparation                      (2) sample application in HPTLC.
  - On the basis of van Deemter equation, explain the effect of longitudinal diffusion on band broadening.
  - What is the difference in the role of mobile phase in liquid chromatography and gas chromatography?

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