

( 3 Hours )

( Total Marks : 100 )

Please check whether you have 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. Attempt **any four** of the following:

- A Describe three mechanisms responsible for the transport of dissolved species to and from the electrode surface in polarography. 5
- B What are the advantages and limitations of a dropping mercury electrode? 5
- C In a polarographic analysis of  $\text{Cd}^{+2}$ , the following results were obtained. 5  
 $i_d = 6.34 \text{ mA}$ ,  $D = 6.2 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$ ,  $m = 2.38 \text{ mg s}^{-1}$ ,  $t = 2.9 \text{ s}$   
 Calculate the concentration of  $\text{Cd}^{+2}$  in the sample solution.
- D The following data were obtained from the polarographic analyses of standard  $\text{Pb}^{+2}$  solutions. 5

$\text{Pb}^{+2} \text{ (mM)}$	0.0	0.51	1.02	2.04	3.06	4.08
$i_d \text{ (}\mu\text{A)}$	1.32	5.65	10.70	19.08	27.91	36.08

Two aqueous samples from the Silver valley area were analysed and gave  $i_d$  of 7.75 and 30.01  $\mu\text{A}$  respectively. What are the  $\text{Pb}^{+2}$  concentrations in these two samples?

- E With the help of a labelled diagram, describe rotating platinum electrode. Give any one of its limitation. 5
- F Explain: a) use of gelatin in polarographic analysis. 3  
 b) with an example any one type of titration curve obtained in amperometric titrations. 2

Q.2. Attempt **any four** of the following:

- A Explain the principle of GC. 5
- B How do gas-liquid and gas-solid chromatography differ? 5
- C Explain flame ionisation detector in GC. Give one advantage. 5
- D The retention times of two components X and Y on column were found to be 3.45 min and 5.16 min respectively. Calculate i) HETP for each peak, ii) number of theoretical plates for each component, if the width of the two peaks is 0.42 and 0.63 min. respectively and the time for mobile phase was 0.18 min. with the length of the column as 0.231 m. 5
- E What is ion exchange chromatography? Give any four ideal properties of resin. 5

- F Define ion exchange capacity. How can the ion exchange principle be applied to demineralisation of water? 5
- Q.3 Attempt **any four** of the following:
- A Discuss pasteurization and pH control as methods of food preservation. 5
- B Write the nutrients present in milk and discuss the detection of any four adulterants in milk. 5
- C Describe Cole's Ferricyanide method to estimate reducing sugars in honey. 5
- D What is the role of chicory in coffee? Discuss the analysis of coffee for caffeine. 5
- E Write the composition of face powder and describe a method to estimate amount of calcium and magnesium in face powder complexometrically. 5
- F Discuss: a) ash analysis of lipstick and 3  
b) the constituents present in antiperspirants 2
- Q.4 Attempt **any four** of the following:
- A Discuss the factors that influence the TG curve. 5
- B Draw a schematic diagram of DTA set up and discuss any three of its components. 5
- C Explain: a) TGA curve of  $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$  and 3  
b) DTA curve of  $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$  2
- D Discuss the application of TGA for the determining the percentage composition of mixture of calcium oxalate and magnesium oxalate. 5
- E Describe the experimental set up for thermometric titrations and explain its application in the titration of boric acid against NaOH. 5
- F Explain the significance of any five parameters w.r.t. validation of analytical methods. 5
- Q.5.A State true or false (**any five**) 5
- a) Oxygen gas is bubbled through the experimental solution prior to polarographic analysis.
- b) Amperometric titrations may lead to inaccurate results due to co-precipitation.
- c) KCl is added as a supporting electrolyte in polarography.
- d) In polarography, DME is used as non-polarisable electrode.
- e) The rotating platinum electrode, can be used at positive potential upto + 0.9V versus SCE.
- f) The diffusion current is directly proportional to the concentration of electroactive species.
- g) A non-polarisable electrode will have its own potential.
- h) The magnitude of diffusion current is also known as wave height.



Q.5.B Select the correct option (**any five**)

5

- a) Theoretical plates are used to \_\_\_\_\_.  
i) estimate the efficiency of a column ii) determine the thickness iii) measure the distribution of the analyte between mobile phase and stationary phase.
- b) In GC, \_\_\_\_\_ is used for electron capture detector.  
i) beta ray source ii) UV lamp iii) mercury vapour lamp
- c) The affinity for the resin increases as the charge on the ion \_\_\_\_\_.  
i) increases ii) decreases iii) remains same
- d) Anion exchange resin is a polymer containing \_\_\_\_\_ groups as an integral part of the resin.  
i) amino ii) sulphonic iii) carboxylic
- e) Smaller the size of the hydrated ion, affinity of the resin for the ions will \_\_\_\_\_.  
i) be more ii) be less iii) remain same
- f) In \_\_\_\_\_ columns a thin layer of adsorbent is coated to the inner walls of the column.  
i) open tubular ii) packed iii) both (i) and (ii)
- g) Band broadening due to concentration gradient within the band is known as \_\_\_\_\_.  
i) Eddy diffusion ii) Longitudinal diffusion iii) Non equilibrium mass transfer

Q.5 C State true or false (**any five**)

5

- a) Salts of sulphur dioxide are used as preservative for fruits.
- b) Deodorants arrest perspiration.
- c) Determination of lactose in milk by Lane Eynon method involves a redox reaction.
- d) Polyphenols are adulterants in tea.
- e) The role of mannitol in determination of boric acid titrimetrically, is to maintain the pH.
- f) Antiperspirants can act as deodorants.
- g) Food treated with irradiation during preservation does not become radioactive itself.
- h) The action of sodium salt of benzoic acid as preservative is pH dependent.

Q.5 D Select the correct option (any five)

5

- a) How many peaks are seen in the DTA curve for dehydration of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ?
  - i) three endothermic      ii) two endothermic
  - iii) three exothermic      iv) two exothermic.
- b) In the application of DTA, which of the following parameters is measured for glasses?
  - i) composition   ii) solubility   iii) cooling temperature   iv) transition temperature
- c) What do the horizontal flat plateaus in the TGA curve indicate?
  - i) weight loss   ii) melting process   iii) no weight loss   iv) enthalpy change
- d) Which of the following process can be studied by DTA?
  - i) phase diagrams   ii) fragmentation of molecule
  - iii) bending vibrations in molecules   iv) none of these.
- e) Which of the following option is appropriate for TGA and DTA?
  - i) TGA and DTA measure only weight
  - ii) TGA measures only weight while DTA measures other factors as well.
  - iii) TGA and DTA measure only temperature.
  - iv) TGA measures only temperature while DTA measures other factors as well.
- f) Which of the following is the essential condition for thermometric titrations?
  - i) enthalpy change      ii) weight change
  - iii) no change in enthalpy   iv) none of these.
- g) Select the false statement from the following?
  - i) Method validation is an integral part of good analytical practice.
  - ii) Method validation can be used to judge the quality, reliability and consistency of analytical results.
  - iii) Method validation is not required for established methods revised to incorporate improvements.