

[Time: 3 Hours]

[Marks:75]

Please check whether you have got the right question paper.

- N.B:**
1. All questions are **compulsory**.
  2. Use of log tables / non- programmable scientific calculator is allowed.

Useful constants:-

$$c = 2.99 \times 10^8 \text{ m s}^{-1} \quad h = 6.626 \times 10^{-34} \text{ Js}$$

$$N_A = 6.023 \times 10^{23} \text{ mol}^{-1} \quad R = 8.314 \text{ JK}^{-1} \text{ mol}^{-1}$$

$$1 \text{ eV} = 1.602 \times 10^{-19} \text{ J} \quad e = 1.602 \times 10^{-19} \text{ C}$$

$$m_e = 9.11 \times 10^{-31} \text{ kg} \quad m_p = 1.673 \times 10^{-27} \text{ kg}$$

$$k = 1.381 \times 10^{-27} \text{ JK}^{-1}$$

**Q.1 Attempt any five of the following.**

- Explain the term ESCA chemical shifts. **03**
- What is diffuse reflectance spectroscopy? **03**
- Point out the main differences between DTA and DSC. **03**
- List the advantages of microelectrodes in voltammetry. **03**
- What are biocatalytic membrane electrodes? **03**
- Distinguish between differential pulse polarography and square wave polarography. **03**
- Why is comparative method preferred over absolute method in radiochemical thermal neutron activation analysis? **03**
- What is the need for hyphenation in trace analysis? Which instruments can be hyphenated? **03**

**Q.2** **a)** Describe the depth profiling of surfaces using Auger electron spectroscopy. **05****OR**

- Explain the working of sample scanners used in scanning tunneling microscope. **05**
  - What is chemiluminescence? Explain the determination of organic species using chemiluminescence. **05**
- OR**
- Explain the term circular dichroism. What are the advantages of CD over ORD? **05**
  - The UPS spectrum of Nitrogen excited by He ( $\lambda=58.43 \text{ nm}$ ) showed kinetic energy of ejected electron equal to 5.64 eV. Calculate the ionization energy of Nitrogen. **05**

- Q.3**
- a) Describe the furnace and sample holders used in TGA. **05**
- OR**
- a) Explain the construction and working of power compensated DSC instrument. **05**
- b) What is an ionophore? Explain the use of ionophores in membrane electrodes. **05**
- OR**
- b) Draw the diagram of the cell used in chronopotentiometry and describe the basic experimental set up. **05**
- c) In the thermo gravimetric analysis of 0.250 g of  $\text{Ca(OH)}_2$  [Mol.Wt. = 74] the Loss in weight at different temperatures was **05**
- i. 0.019 g at 373 – 423 K [loss of hygroscopic water]
  - ii. 0.037 g at 773 – 833 K [dehydration]
  - iii. 0.036 g at 1173 – 1223 K [dissociation]
- Determine the composition of  $\text{Ca(OH)}_2$  in the sample.
- Q.4**
- a) **Attempt any two of the following.**
- i) What is pulse polarography? Draw the pulse polarogram. Why quality of polarographic analysis is enhanced in pulsed polarography than I normal polarography? **05**
  - ii) What is stripping voltammetry? What are its types? Explain the steps involved in the technique. **05**
  - iii) Explain how organic synthesis is carried out using electrochemical methods with suitable examples. **05**
  - iv) Describe the characteristic features of AC polarography. **05**
- b) The diffusion current due to  $\text{Cu(II)}$  ions in 5 mL of solution was  $13.8 \mu\text{A}$  on a DC polarogram. When 0.1 mL of  $1 \times 10^{-3} \text{ M}$   $\text{Cu(II)}$  ions solution was added to the original solution, the new current was  $28.6 \mu\text{A}$ . Calculate the concentration of  $\text{Cu(II)}$  ions in the sample. **05**
- Q.5 Attempt any Three of the following.**
- a) What is gamma ray radiography? What are its applications? **05**
  - b) Explain the technique of substoichiometric isotope dilution analysis. **05**
  - c) Describe the basic experimental set up for the spectroelectrochemical experiment. **05**
  - d) Calculate the activity of 25 mg sample of an Aluminium alloy containing 0.046% of Manganese after irradiation for 0.5 hr in a flux of  $5 \times 10^{13} \text{ n cm}^{-2} \text{ s}^{-1}$  **05**  
(Given:  $t_{1/2}$  of  $^{56}\text{Mn} = 100 \%$ )
  - e) Describe the Thermospray interface used in LC – MS technique. **05**