D.K.M. COLLEGE FOR WOMEN (AUTONOMOUS), VELLORE-1

MODERN INSTRUMENTATION TECHNIQUES (ELECTIVE-I)

UNIT-I POLAROGRAPHY AND AMPEROMETRY

Section-A (6 Marks)

1. Polarography is a highly elegant technique invented by Jaroslav Heyrovsky for which he received the Nobel Prize in 1959. (i) What is the unique feature of polarography which separates it from other electroanalytical techniques? (ii) Sketch a polarogram and label two important details of the polarographic wave on your diagram.
2. Electrochemical or electrolytic reactions at electrodes are categorised into reversible and irreversible reactions. (i) Why does an electrochemical process appear irreversible? (ii) Explain with a sketch how the polarographic current-potential curve characterising an electrochemical process of reduction changes when the process becomes irreversible. (iii) What is the potential value in mV of the separation between the anodic and cathodic peaks of a reversible one electron cyclic voltammogram?
3. Discuss the following different currents are possible in polarography: 1.Migration current/residual current: 2.Diffusion current 3.limiting current 4.Catalytic current 5.Adsorption current
4. How the Modern polarography is classified? Explain NPV, DPV, SWV and CV?
5. What is the key experimental feature that made the invention of DC polarography by J. Heyrovsky highly significant for its time.
6. Define the term polarogram and polarography
7. What are the advantages of DME
8. Write about polarographic maxima
9. Give the potential range of DME
10. What are the causes of migration current

11. How can migration current can be eliminated
12. What do you meant by diffusion current
13. Distinguish between limiting current and diffusion current
14. What is referred to as capillary characteristics and what are the factors affecting them
15. In what range should the temperature be kept during polarographic experimentand why?
16. What is the polarographic equation and what does it represent?
17. How can you determine whether a reaction is reversible or not with the help of "Equation of the polarographic wave"?
18. What gives rise to adsorption waves?
19. Give reasons for the addition of a small concentration of a surface active substanceduring the measurement of true diffusion current.
20. Draw the basic apparatus for polarographic analysis
21. Give any two advantages of amperometry.
22. Give three reasons for using a dropping mercury electrode in polarography.
23. Define half-wave potential, diffusion current, kinetic current, catalytic currentand residual current
24. Why is a high supporting electrolyte concentration used in mostelectroanalytical procedures?
25. What is the effect of complexing agent on the reduction of metal ion inPolarography?
26. Derive polarographic equation.
27. Write Ilkovic equation and define the terms
28. On what factors does the migration current depend on? Explain with the help of an equation.
29. For what have the adsorption waves been used?
30. Explain the method of standard addition in quantitative polarographic analysis?

31. Explain the procedure of determination of Zn with EDTA with the help of amperometry.
32. Is amperometric titration capable of greater accuracy than non-titrativepolarographic method?
33. Explain different types of titration curves obtained in amperometry giving examples.
34. A rotating platinum electrode is preferable over DME in which situation?

Section-B (15 Marks)

35. Give the titration curve expected when Fe (II) is titrated with Ce (IV) solution in acid medium using two platinum electrodes.
36. Define the components of a simple polarography
37. If several different complexing agents are present in the solution, how will this affect the polarographic behaviour?
38. Linear sweep and CV waveforms are implemented in modern instruments using a digital waveform, Draw the input waveform and the typical output for a reversible solution reaction.
39. Describe the shape and the main quantitative characteristics of cyclic voltammograms of a surface confined redox species, e.g. O(surf) + e = R(surf) at < 50 mV/s for films ~15 nm thick that follows the reversible, ideal thin film voltammetry model. Indicate approximate positions of $E^{o'}$ on the CV, how peak current depends on scan rate, and how the amount of O(surf) on the electrode surface can be measured from CV.
40. List and briefly explain the advantages of - electrochemical detectors for liquid chromatography - microelectrodes - pulse voltammetric techniques - stripping voltammetry - cyclic voltammetry - modified electodes
41. How is irreversibility recognized (diagnosed) in cyclic voltammetry?
42. Does O_2 need to be accounted for in this experiment? Why or why not? Why is cyclic voltammetry an unstirred voltammetry technique? Why is a triangular waveform used as the excitation waveform in cyclic voltammetry rather than a square waveform?

43. What reaction will occur if a solution containing Pb^{2+} is added to a solution containing MgY^{2-}? What reaction will occur if a solution containing Ba^{2+} is added to a solution containing CuY^{2-}? Calculate the fraction of EDTA present as Y^{4-} in solution at the following pH: a) pH 8. 0 b) pH 11.0.
44. A 100.0 mL aliquot of city drinking water was treated with a small amount of an ammonia – ammonium chloride buffer to bring the pH to 10. After the addition of Calmagite indicator the solution required 21.46 mL of 5.140 ×10-3 M EDTA for titration. Calculate the hardness in terms of parts per million calcium carbonate.
45. A standard solution of EDTA (0.100 M) is being used to titrate 25.00 ml of a 0.100 M Zn^{2+} solution (buffered at pH 8). Calculate the value of pZn after the addition of: a) 0 ml of EDTA solution. b) 10.00 ml of EDTA solution c) 25.00 ml of EDTA solution d) 50.00 ml of EDTA solution.
46. For some titrations with EDTA, it is necessary to use a NH_3/NH_4^+ buffer. However, the NH_3 can form complexes with many metals – for example, Zn^{2+} . a) Would you expect the presence of NH3 to have any effect on the value of pZn? Briefly explain. b) Would you expect the presence of NH3 to affect the shape of the titration curve? Briefly explain.
47. What would happen if the metal-indicator complex was so stable that the EDTA could not remove the metal from the complex?
48. Write a short note on masking and demasking agents, and explain the different types of EDTA titrations
49. Discuss the different types of waves obtained during polarography and amperometric titrations
50. Discuss the unique applications of the polarography, amperometric and complexometric titrations

UNIT – II CHROMATOGRAPHIC TECHNIQUES

Section-A (6 Marks)

1. Explain the Adsorption, partition Chromatography.

2. Explain the detectors in GLC.
3. Discuss the application of GLC.
4. Write note on HPTLC – Principle, instrumentation and applications.
5. Explain the pumping system in HPLC.
6. Give an account on Gel chromatography and ion exchange methods.

Section-B (15 Marks)

7. Explain in detail about the Instrumentation and principle of GLC.
8. Explain in detail about the Instrumentation and principle of HPLC.
9. Discuss the Adsorption and partition and ion exchange chromatography.
10. i. Write a short note on Thermal Conductivity, Flame ionization and Electron capture (7)

    ii. Give an account on HPTLC – Principle, instrumentation and applications. (8)

UNIT-III RADIO ANALYTICAL CHEMISTRY AND ATOMIC ABSORPTION SPECTROSCOPY

Section-A (6 Marks)

1. Explain in detail about the TGA and DSC.
2. Explain in detail about DTA.
3. Explain in detail about ICP.
4. Give an account for Isotope dilution analysis.
5. Discuss in detail about radiometric titrations.
6 . Write a short note on Radio immunoassay.
7. Write a short note on Neutron activation analysis.

Section-B (15 Marks)

8.Explain in detail about the atomic absorption spectroscopy.

1. Describe in detail about radiometric titrations and Atomizers.

UNIT -IV

ELECTROGRAVIMETRY, COULOMETRY,POTENTIOMETRY AND CONDUCTOMETRY

Section-A (6 Marks)

1. Explain about decomposition potential and its significance.

2. Explain about over voltage and its significance.
3. Explain a method to determine the Nickel and gold in elemental analysis.

Section-B (15 Marks)

4. Explain in detail about the principle and instrumentation and applications of coulometry analysis.
5. Explain in detail about the principle and instrumentation and applications of conductometric titrations.
6. Explain in detail about the principle and instrumentation and applications of potentiometric titrations.

UNIT – V NANOTECHNOLOGY

Section-A (6 Marks)

1. What is nanotechnology? How big is nonaometer? What are zero, one, two and three dimensional nanomaterials?
2. How does nanotechnology work? What is so special about nanotechnology?
3. Write a note on nanomaterials, graphene?
4. Where is nanotechnology being developed?
5. Where will nanotechnology take us? Are they any specific health or other risks from nanoproducts?
6. How does AFM works?
7. What kind of samples can be analysed by AFM? What are the applications of AFM?

Section-B (15 Marks)

8. What kind of artifacts can occur in AFM images? How can you avoid getting artifacts in your images?
9. Discuss the application of AFM, STM, SEM and TEM
10. Explain in detail about the nanosensors:
    A).electrochemical sensors, b). Nanobiosensors.

11. How the nanomaterials plays a role in biomedical field, optics and electronics. Discuss the functions of FIB, AFM, DPN and SPL
12. Write a detailed note on optical microscopies
13. How scanning nearfield optical microscopy plays a major role than the particle size analysis