## P.G. DIPLOMA IN ANALYTICAL CHEMISTRY (PGDAC)

## Term-End Examination December, 2011

00816

## MCH-004 : ELECTROANALYTICAL AND OTHER METHODS

Time: 3 hours Maximum Marks: 75

**Note:** Attempt any five questions. All questions carry equal marks.

- 1. (a) Derive Nernst equation for the chemical reaction  $M^+ + e^- \rightleftharpoons M$ 
  - (b) Explain the basis of classification of electroanalytical methods. Describe each method in 2-3 lines.
  - (c) How electrode potential measurement can 2+3 be used for the determination of equilibrium constant (k)? Calculate the value of k for the reaction.

$$Ce^{4+} + Fe^{2+} \rightleftharpoons Ce^{3+} + Fe^{3+}$$

Given that  $E^{\circ} \text{ Ce}^{4+}/\text{Ce}^{3+} = 1.70\text{V}$ ,

$$E^{\circ} \text{ Fe}^{2+}/\text{Fe}^{3+} = 0.771 \text{ V}$$

- 2. (a) Explain the working of a glass membrane electrode. How can it be used for the determination of pH?
  - (b) Explain why it is advisable to use first and second derivative curves for the identification of end point in a pH titration. How do you perform the titration of acetic acid and NaOH?

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- (c) What do you understand by ionic mobility 5 and transport number. List various factors affecting conductivity of solution.
- 3. (a) Discuss advantages and limitations of conductivity measurements. Explain how conductance measurement of soil helps in classifying the soil as saline and non saline.
  - (b) What is polarization? Explain concentration polarization and kinetic polarization. Discuss the importance of over voltage.
  - (c) What is coulometry? Write its advantages over electrogravimetry. Describe a method of separation of nickel and cobalt by controlled potential coulometer.
- 4. (a) What is polarography? Explain the 5 principle of DC polarography and draw the nature of sampled DC polarogram depicting  $E_{1/2}$ .

	(b)	voltammetry (CV). Draw a schematic diagram showing instrumentation explaining its components. Why CV has been recognised as the most versatile electrochemical technique?	5
	(c)	Write Ilkovic equation and explain all the terms. Show that diffusion current $(i_d)$ is proportional to the concentration and a plot between $i_d$ and $c$ can be used for the determination of concentration.	5
5.	(a)	Describe various factors affecting the diffusion current.	5
	(b)	Describe the following briefly  (i) Polarographic maxima and its suppression  (ii) Biamperometry and its applications	. 5
	(c)	In coulometric titration of iodine with 20 cm <sup>3</sup> of thiosulphate, 25 minutes were required to oxidise thiosulphate and 200 mA of current were used. Calculate the molarity of thiosulphate solution.	5
6.	(a)	Write essential differences between DTA and DSC techniques.	5
-	(b)	What is thermogravimetry? Draw a labelled schematic diagram of a thermobalance and describe all its	5

components briefly.

- (c) Draw the nature of thermogravimetric curve 2+3 for a compound undergoing decomposition in two stages. Calculate percent mass loss in following cases -
  - (i)  $CaC_2O_4 \rightarrow CaCO_3 \rightarrow CaO$
  - (ii)  $Ca(OH)_2 \rightarrow CaO$
  - (iii)  $CuSO_4 5H_2O \rightarrow CuSO_4 \rightarrow CuO$
- 7. (a) Explain characteristics of thermometric titrations (TT). What type of information is derived from an enthalpogram. In what respects these differ from convential titrations.
  - (b) Name three processes by which gamma rays interact with matter. Explain these briefly.
  - (c) Explain the principle of neutron activation analysis (NAA) and how is it used for the quantitative analysis of a sample.
- 8. (a) Describe various sources of background activity of a counter. How these are eliminated or minimized.
  - (b) How many  $\alpha$  and  $\beta$  particles will be emitted during decay of  $^{234}$ U to  $^{214}$ Bi.
  - (c) Explain why GM counter cannot be used for recording gamma ray spectrum.
  - (d) Capillary height should not be disturbed during recording of polarogram or else capillary characteristics will change.

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(e) Thermogravimetric analysis is considerably affected by the furnace atmosphere i.e inert or oxygen and useful information about decomposition reaction may be obtained.