# LOYOLA COLLEGE (AUTONOMOUS), CHENNAI – 600 034

# M.Sc. DEGREE EXAMINATION - CHEMISTRY

## FIRST SEMESTER - APRIL 2010

### **CH 1809 - ANALYTICAL CHEMISTRY**

Date & Time: 30/04/2010 / 1:00 - 4:00 Dept. No. Max. : 100 Marks

### PART A

# Answer all the questions.

 $10 \times 2 = 20$ 

- 01. What are random errors? Give an example.
- 02. Round each of the following numbers to three and two significant figures.
  - a) 1.007

b) 28755

- 03. What is the principle of DSC?
- 04. Give any one method of packing the column in HPLC.
- 05. The standard deviation from one set of 11 determinations was SA = 0.210, while standard deviation from another 13 determinations was SB = 0.641. Is there any significant difference between the precision of these two sets of results? (F  $_{10\%} = 2.28$ ).
- 06. What is replacement titration? Give an example.
- O7. Calculate the equilibrium constant for the reaction
  CH<sub>3</sub>COOH + OH<sup>-</sup> ↔ CH<sub>3</sub>COO<sup>-</sup> + H<sub>2</sub>O and K<sub>a</sub> of CH<sub>3</sub>COOH is 1.8 x 10 <sup>-5</sup>.
- 08. What is polarographic maxima?
- 09. Calculate the current needed to deposit all nickel in 100 ml of 0.02M NiSO<sub>4(aq)</sub> in 60 minutes?
- 10. What is selectivity coefficient of an ion with reference to an ion selective electrode?

## PART B

## Answer any eight questions.

 $8 \times 5 = 40$ 

- 11. Analysis of an alloy of iron gave the following percentages for the iron content 7.08, 7.21, 7.12, 7.09, 7.16, 7.14, 7.07, 7.14, 7.18, 7.11. Calculate the mean, standard deviation and variance.
- 12. Describe the factors affecting TGA.
- 13. What are the important characteristics of a good detector in HPLC? Explain the types of detectors used.
- 14. Draw and explain the working principle of katharometer.
- 15. How is sulphate determined by turbidimetry?
- 16. Draw the TGA and DTG curves of CuSO<sub>4</sub>.5H<sub>2</sub>O and explain.
- 17. 0.3953 g of sample containing only NaCl and NaBr is dissolved in water consumed 49 ml of 0.1M AgNO3 solution. Calculate the percentage of NaCl and NaBr in the sample. Also express the answer in ppm of NaCl.
- 18. Discuss the principle of cyclic voltammetry with a suitable diagram.
- 19. Derive an expression for pH of aqueous NaHCO<sub>3</sub> and calculate the pH of a solution obtained by mixing 20 ml of 0.1M Na<sub>2</sub>CO<sub>3</sub> with 20 ml of 0.1M HCl.  $K_1$  and  $K_2$  for H<sub>2</sub>CO<sub>3</sub> are 4.2 x 10  $^{-7}$  and 4.8 x 10  $^{-11}$ .

- 20. Explain the factors affecting the equivalence point in complexometric titrations involving EDTA.
- 21. What is the role of i) dextrin in precipitation titrations and ii) supporting electrolyte in polarography.
- 22. What is the principle of coulometric titrations? Explain with an example.

## **PART C**

# Answer any four questions.

 $4 \times 10 = 40$ 

- a) Discuss the sample injection system and derivatisation of samples in Gas chromatography.b) Explain the principle and instrumentation of capillary electrophoresis. (5+5)
- 24. Describe the working principle and instrumentation involved in atomic absorption spectroscopy.
- a) A 0.525g of an alloy steel is dissolved, Mn is oxidized to permanganate and the solution is diluted to 100 ml in a volumetric flask. The absorbance at 525 nm in 1 cm cell is 0.496. The molar absorptivity of permanganate is 2.24 x 10<sup>3</sup>. Calculate the percentage of manganese in steel.
  - b) Explain the different types of pumps used in HPLC for solvent delivery system. (6+4)
- 26. a) Discuss the factors that influence  $\triangle pM$  the equivalence point in precipitation titrations.
  - b) 3g sample of phenol and other inert materials was dissolved in ethylene diamine and titrated with 0.04M sodium aminoethoxide requiring 16.4 ml to reach the end point. Calculate the percentage of phenol. (6+4)
- 27. a) Explain how the percentage composition of Mg<sup>2+</sup>, Zn<sup>2+</sup> and Hg<sup>2+</sup> in a solution be determined using EDTA titrations with special emphasis on the principles.
  - b) Show that the potential at the equivalence point of

$$MnO_4^- + Fe^{2+} + H^+ \rightarrow Mn^{2+} + Fe^{3+} + H_2O$$
 is pH dependent. (6+4)

- 28. a) Gallium(III) forms a complex with an organic ligand (L). The reaction at the DME in polarography is  $GaL_x + 3e^- \rightarrow Ga+ xL$  (charges omitted). A graph of  $E_{1/2}$  (y-axis) vs log[L] gave a straight line with slope = -78.7 mV. Calculate 'x'.
  - b) Explain diverse ion effect with an example. (5+5)

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