# LOYOLA COLLEGE (AUTONOMOUS), CHENNAI – 600 034

#### M.Sc. DEGREE EXAMINATION - CHEMISTRY

### FIRST SEMESTER - NOVEMBER 2007

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

**AD 17** 

Date: 02/11/2007 Time: 1:00 - 4:00 Dept. No.

Max.: 100 Marks

#### PART - A

## **Answer ALL the questions:**

 $(10 \times 2 = 20)$ 

- 1. An analyst reports the percentage of carbonate in 80% pure CaCO<sub>3</sub> as 50%. Calculate the absolute error and relative error in ppt.
- 2. Mention the two important conditions to apply the method of least squares for drawing the calibration graph.
- 3. Explain back titration in complexometry with an example.
- 4. Mention the equivalence point potential for  $B^{+2}$  vs.  $A^{+3}$ ; given

$$A^{3+} + e^{-} \rightleftharpoons A^{2+} \quad E^{o} = + 1.42 \text{ V}$$
  
 $B^{4+} + 2e^{-} \rightleftharpoons B^{2+} \quad E^{o} = + 0.40 \text{ V}$ 

- 5. Mention the advantages of HPLC over GLC.
- 6. What is a hollow cathode lamp?
- 7. State the principle in the spectrophotometric analysis of iron(III).
- 8. Mention any two applications of DTA.
- 9. Mention any two advantages of DME.
- 10. Sketch a cyclic Voltamonogram for a reversible system. Explain the parameters.

## PART – B

## Answer any EIGHT questions:

 $(8 \times 5 = 40)$ 

- 11. K<sub>a</sub> for HA is 10<sup>-9</sup>. Check whether HA can be estimated by titration against OH<sup>-</sup>. Explain.
- 12. pH at the equivalence point for HCl vs OH<sup>-</sup> is 7, while it is around 8 for CH<sub>3</sub>COOH vs OH<sup>-</sup>. Explain.
- 13.  $K_{a_1}$ ,  $K_{a_2}$ ,  $K_{a_3}$ , and  $K_{a_4}$  for EDTA are  $10^{-2}$ ,  $10^{-3}$ ,  $10^{-8}$  and  $10^{-12}$  respectively. Determine the concentration of completely ionized form of EDTA in  $10^{-2}$  M aq. EDTA at pH = 12.
- 14. 0.2535g sample of sodium oxalate consumed 40.20mL of 0.0730N KMnO<sub>4</sub>. Determine the percentage of sod. oxalate in the sample and express it scientifically.
- 15. Discuss the principle involved in GLC. Name two liquids, which can be used, as immobile phase.
- 16. Show that the equivalence point potential for the titration of Fe<sup>+2</sup> vs. KMnO<sub>4</sub> is pH dependent.
- 17. How is Copper determined by electrogravimetry method?
- 18. Discuss the deviations from Lambert-Beer's Law.
- 19. How is sulphate determined by turbidimetric method?
- 20. Discuss any two applications of spectrocourimetric method of analysis.
- 21. Discuss the thermo gravimetry (TG) of Ca(OOC-CH<sub>3</sub>)<sub>2</sub>.H<sub>2</sub>O.
- 22. How is Faraday's Law useful in Coulometric analysis?

#### PART - C

### Answer any FOUR questions:

 $(4 \times 10 = 40)$ 

- 23. One common way to determine phosphorus in urine is to treat the sample, after removing the protein, with molybdenum(VI) and then reduce the resulting
  - 12-Molybdophosphate complex with ascorbic acid to give an intense blue coloured species called molybdenum blue. The absorbance of molybdenum blue can be measured at 650nm. A patient produced 1122 mL of urine in 24 hours.

A 1mL aliquot of the sample was treated with Mo(VI) and ascorbic acid and was diluted to a volume of 50.00mL. A calibration curve was prepared by treating 1.00 mL aliquots of phosphate standard solutions in the same manner as the urine sample. The absorbance of the standard and the urine sample were obtained at 650 nm and the following results were obtained.

Solutions/ppm	Absorbance
P	
1.0	0.230
2.0	0.436
3.0	0.638
4.0	0.848
Urine sample	0.518

- a) Determine the slope and intercept.
- b) Calculate the ppm of P in the urine sample.
- c) What is  $S_r$  if the absorbance of the urine sample is the average of 3 determinations.
- 24. Draw the flow sheet diagram of HPLC and explain the principle and its working.
- 25. a) Explain the factors, which influence  $\Delta pM$  at the equivalence point. (4)
  - b) Calculate the concentrations of all the species in a mixture containing 75.00mL of 0.025M Fe<sup>+3</sup> and 50.00mL of 0.00375M EDTA at pH<sub>2</sub>.

Fe<sup>3+</sup> + Y<sup>4-</sup> 
$$\rightleftharpoons$$
 FeY<sup>-</sup>  $K_{abs} = 10^{25}$   
The value of  $\alpha$  at pH<sub>2</sub> is  $10^{-14}$ . (6)

- 26. Describe the instrumentation and method of analysis by atomic absorption analysis.
- 27. Write the Ilkovic equation. Explain the terms. How are Cu<sup>2+</sup> and Cd<sup>2+</sup> analysed simultaneously by polarographic method.
- 28. Discuss the factors that influence TG-DTA curves.

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