LOYOLA COLLEGE (AUTONOMOUS), CHENNAI – 600 034

M.Sc. DEGREE EXAMINATION – CHEMISTRY

FIRST SEMESTER – NOVEMBER 2009

CH 1809 - ANALYTICAL CHEMISTRY

Date & Time: 11/11/2009 / 1:00 - 4:00   Dept. No.   Max. : 100 Marks

PART A

Answer all the questions $10 \times 2 = 20$

1. What are gross errors? Give an example.

2. What is the principle of GLC?

3. Mention any two functions of burner in flame photometer.

4. Define gradient elution.

5. Draw and explain the DTA curve of sulphur.

6. What mass of KIO_4 is needed to prepare 100 ml of 0.5N solution of it as per the redox reaction

    $IO^{4-} + I^- + H^+ \longrightarrow I_2 + H_2O$ (unbalanced)

7. What is the role of dextrin in the titration of Ag^+ with $Ce^-_{(aq)}$?

8. Calculate the amount of Sb^{3+} present if 2.476 min was needed to coulometrically titrate Sb^{3+} in a solution with 3.5 mA current forming Sb^{5+}.

9. What is the pH of 0.05M NaHA if $K_1 = 10^{-5}$ and $K_2 = 10^{-8}$ for $H_2A_{(aq)}$?

10. Write Ilkovic equation. Mention the terms involved.

PART B

Answer any eight questions $8 \times 5 = 40$

11. Analysis of a sample of an ore gave the following percentage values for the iron content: 7.08, 7.21, 7.12, 7.09, 7.16, 7.14, 7.07, 7.14, 7.18, 7.11. Find out the mean, standard deviation and variance for the values.

12. Briefly explain the various sample injection systems used in HPLC.

13. What is TCD? Describe its working principle.

14. Explain the DTG analysis of $CuSO_4.5H_2O$.

15. What are the factors affecting fluorescence emission? Explain.

16. Write short notes on the chemical interferences in FES.

17. Draw and explain the need of atomizer unit in AAS.

18. A 3g sample containing phenol and other inert material was dissolved in ethylenediamine and titrated with 0.04M sodium aminoethoxide requiring 16.4 ml to reach the end point. Calculate the ppm of phenol in the sample as per the reaction given below.

    $C_6H_5OH + NaOCH_2CH_2NH_2 \longrightarrow C_6H_5ONa + HOCH_2CH_2NH_2$

19. 50 ml of 0.01M Ca^{2+} is titrated with 0.01M EDTA at pH 10. Calculate pCa at a) 50 ml, b) 60 ml addition of EDTA. K_{abs} of CaY^{2-} is 5×10^{10} and $\alpha 4$ at pH 10 $= 0.35$.

20. Derive an expression for the potential at the equivalence point for the redox reaction

    $Cr_2O_7^{2-} + 14H^+ + 6Fe^{2+} \longrightarrow 2Cr^{3+} + 6Fe^{3+} + 7H_2O$.

21. Explain the principle of controlled potential coulometry.

22. Calculate the relative error when 50 ml 0.1M NaCl is titrated with 0.1M $AgNO_3$ using 2×10^{-3}M K_2CrO_4 solution as indicator. Assume that the titration is stopped at the very onset of $AgCrO_4$ precipitate formation. K_{sp} of AgCl, Ag_2CrO_4 are 1.8×10^{-10}, 1.2×10^{-12} respectively.

PART C
Answer any four questions   4 x 10 = 40

23. a) Draw and explain the working principle of TG.
    b) How is phosphate determined by turbidimetry?   (5+5)

24. Briefly explain the principle and working nature of capillary electrophoresis.

25. a) Discuss any two types of spectrophotometric titrations with examples.
    b) A 0.525g sample of an alloy steel is dissolved, the manganese is oxidized to permanganate and the solution is diluted to 100ml in a volumetric flask. The absorbance at 525 nm in a 1.00 cm cell is 0.496. The molar absorptivity of MnO_4^- is 2.24×10^3. Calculate the percentage of Mn in the steel. (The molar absorptivity of MnO_4^- is same as Mn^{7+}).Atomic weight of Mn^{7+} is 54.94.   (5+5)

26. a) Explain the principle of cyclic voltammetry.
    b) A solution containing 75 mg of Cu was electrolyzed at 0.25A current for 10 min causing deposition of Cu on Pt cathode. Calculate the percentage of Cu^{2+} remaining in the solution after 15 min.   (7+3)

27. Explain any two of the following:
    a) Characteristics of DMA
    b) Volhard's method
    c) Metallochrome indicators
    d) Enzyme ion selective electrode   (5+5)

28. a) Calculate the molar solubility of CaF_2 in 0.002M HCl solution. K_{sp} of CaF_2 is 1.7×10^{-10} and K_a of HF is 6.9×10^{-4}.
    b) A 24 hour biological specimen was diluted to 200 ml and buffered to pH 10. A 10 ml aliquot was titrated with 30 ml of 0.004M EDTA. The calcium in a second 10 ml aliquot was isolated as CaC_2O_4 (s) redissolved in acid and titrated with 13 ml of the EDTA solution. Assuming that 15 to 200 mg of Mg^{2+} and 50 to 300 mg Ca^{2+} per day are normal, did this specimen fall within these ranges?   (4+6)

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