LOYOLA COLLEGE (AUTONOMOUS), CHENNAI – 600 034

M.Sc. DEGREE EXAMINATION – **CHEMISTRY**

FIRST SEMESTER – **NOVEMBER 2018**

16/17/18PCH1MC04 – ANALYTICAL CHEMISTRY

Date: 01-11-2018   Dept. No.   Max. : 100 Marks
Time: 01:00-04:00

Part-A

Answer ALL questions. $(10 \times 2 = 20)$

1. Calcium oxide content was determined in a calcite sample and gave the following results 56.04, 56.08, 55.95, 56.00 and 56.23%. Apply the Q-test to check if the value 56.23 can be discarded or not. (Q cri=0.71).

2. Highlight the significances of correlation coefficient.

3. Define isocratic and gradient elution.

4. Write the advantages of fused silica capillary column compared with glass or metal columns.

5. Define autoprotolysis constant.

6. How do solubility product and common ion effect influence precipitation titration?

7. In a coulometric titration of 20 mL of $K_2Cr_2O_7$ with iron (III) which is generated in solution took 25 minutes to reduce when 200 mA of current was used. What is the normality of $K_2Cr_2O_7$?

8. How does hydrogen over potential influence electrogravimetric determination of metals?

9. Why a low temperature flame is used for the analysis of alkali and alkaline earth metals in AAS?

10. State Beer-Lambert law.

Part-B

*Answer any **EIGHT** questions.*   **(8 x 5 = 40)**

11. Write a short note on normal error curve and its importance.

12. A chemist reported the following percentage of Cd in a sample 0.084, 0.089, 0.079. 0.085. Calculate the 99% confidence interval of the mean (t=4.604).

13. Write the principle of TLC. How would you identify a compound on a chromatogram?

14. Describe the working principle of thermal conductivity detector with a diagram.

15. Write Van Demeter equation and explain the terms involved in it.

16. How many mL of a 0.1M HCl are required to react completely with 1g mixture of Na_2CO_3 and $NaHCO_3$ containing equimolar amounts of both?

17. Explain the reactions involving glacial acetic acid as non aqueous titrations.

18. Draw and explain the DTA and TGA thermograms of calcium oxalate monohydrate.

19. Explain the principle of controlled potential coulometry.

20. Write an account on the back/counter polaraisation in electrogravimetry.

21. What are the principle of nephelometry and turbidimetry?

22. How is lead content in petrol determined by AAS?

Part-C

Answer any **FOUR** *questions.*   **(4 x 10 = 40)**

23. a) What are systematic errors? and how can we minimize these type of errors?   (7)

   b) In the analysis of sample of iron ore, the following results are obtained $\bar{x} = 10.52$, s = 0.05,   n = 10. The NIST value for this sample is 10.06% Fe. Identify whether the result is significant or not applying t-test? (t = 2.262)   (3)

24. Discuss the principle, instrumentation and applications of capillary electrophoresis.

25. a) Discuss the type of indicators with suitable examples.   (5)

   b) Explain the different ways of calculation of equivalent weights with suitable examples.

                                                                           (5)

26. a) What is the half cell potential at the end point in the titration of Fe (II) with $KMnO_4$ in a H_2SO_4 media if at the end point pH is 2.3. Given that $E°_{Fe^{3+}} = 0.771V$ $E°_{MnO_4-} = -1.51V$. (6)

   b) Mention the advantages of coulometric titrations.   (4)

27. a)Describe the thermogravimetic analysis of $CuSO_4.5H_2O$.   (5)

   b) What is spectrophotometric titration? How is Fe(III) is determined using   spectrophotometry.

                                                                           (5)

28. a) Explain the principle of flame emission spectrometry.   (5)

   b) How is codeine-morphine mixture determined by fluorimetry?   (5)

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