# Reg. No.

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## Third Semester M.Sc. Degree Examination, December 2018 ANALYTICAL CHEMISTRY (CBCS : 2016-17 Syllabus) Principles of Analytical Chemistry

Time : 3 Hours

**Instructions** : Answer Part – **A** and **any four** from Part – **B**. Figures to the **right** indicate marks.

### PART – A

- 1. Answer all the following sub-divisions :
  - a) Explain the indicator action of a redox indicator, diphenylamine in the titration of Fe<sup>2+</sup> versus K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution.
  - b) Suggest a suitable indicator for the titration of 0.1 M acetic acid with 0.1 M NaOH and 0.01 M HNO<sub>3</sub> with 0.01 M NaOH. Give the reason for your choice.
  - c) Why NaOH and EDTA solutions are not primary standards ?
  - d) Write the indicator action of fluorescein in the titration of Cl<sup>-</sup> solution with Ag<sup>+</sup> solution.
  - e) The standard potential for the half-reaction,  $M^{4+} + 2e \rightarrow M^{2+}$ . Is  $M^{2+}$  a good or poor reducing agent ? Justify.
  - f) If 100 mL of water sample is titrated with 26.58 mL of 0.01 M EDTA to Eriochrome Black-T indicator end point, calculate the hardness of water interms of CaCO<sub>3</sub> (Molecular weight of CaCO<sub>3</sub> = 100).
  - g) Why EDTA is commercially available in the form of disodium salt of EDTA ? Write the structure of EDTA.
  - h) What is precipitation from homogeneous solution ? Mention one of its advantages.
  - i) Calculate the potential at the equivalence point in the titration of Sn(II) with Ce(IV) (Given :  $E^{0}Ce^{4+}/Ce^{3+} = 1.44$  V and  $E^{0}$  Sn<sup>4+</sup>/Sn<sup>2+</sup> = 0.15 V).

(9×2=18)



Max. Marks: 70

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#### PART – B

Answer any four full questions.

- 2. a) Explain with chemistry, the determination of nitrogen and sulphur, by acid-base titration.
  - b) How are phenols determined ? Discuss with suitable examples.
  - c) Sketch the titration curve involving the titration of a strong acid with a strong base. (5+5+3=13)
- 3. a) Discuss the steps involved in the determination of carbonates and carboxylic acid functional group.
  - b) List out the solvents used as non-aqueous solvents. Discuss the typical applications of non-aqueous titrations with suitable examples. What are the limitations of non-aqueous titrations ? (6+7=13)
- 4. a) Propose a scheme for the determination of any 3 metal ions in a mixture by complexometric titration using the principle of masking and demasking.
  - b) A 0.5g bronze sample containing Zn is dissolved in 100 mL of acid. If an aliquot of 20 mL need 10.1 mL of 0.011 M EDTA, calculate the percent of Zn in the bronze sample. (Atomic weight of Zn = 65.38 amu)
  - c) Why EDTA titrations are called complexometric titrations ? Explain the indicator action of metallochromic indicator. (5+4+4=13)
- 5. a) Explain any three types of performing EDTA titrations. Give one example for each type.
  - b) The chloride in a 0.6025 g sample of chloride salt is precipitated as AgCl by adding excess AgNO<sub>3</sub> solution. The precipitate is filtered, washed, dried and found to weigh 0.7134 g. Calculate the % chloride in the sample (Atomic masses of Ag and Cl are 107.9 and 35.45, respectively).
  - c) Describe with chemistry the Volhard's method of determination of chloride in a water sample using Ag<sup>+</sup> solution. (5+4+4=13)

(4×13=52)

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- 6. a) What is Karl Fischer (KF) reagent ? Illustrate its application in the determination of traces of water in an organic solvent.
  - b) What is co-precipitation ? List the causes for co-precipitation.
  - c) A 2.55 g coal sample was taken in a crucible weighing 19.35 g. After heating in an electrical oven at 105-110°C for 1 hour, the crucible weighed 21.8 g. The residue was then ignited at 700-750°C to a constant weight when the crucible weighed 19.6g. Calculate the % ash and moisture contents of the coal sample. (5+4+4=13)
- 7. a) A 0.20 g of haematite ore was brought to solution (100 mL) upon acid treatment. A 25 mL was reduced to Fe(II) and consumed 15.0 mL of 0.1N  $K_2Cr_2O_7$  for diphenylamine end point. Calculate the amount of iron present in the given iron sample (Given : Equivalent weight of Fe = 55.85 amu).
  - b) Discuss the conditions for precipitation.
  - c) From the following reactions, give the relation between Normality (N) and Molarity (M) of the titrant. Indicate which type of titrations are these ?

i) 
$$2 S_2 O_3^{2-} + I_2 \rightarrow S_4 O_6^{2-} + 2 I^-$$
 and

ii)  $2 \text{ MnO}_4^- + 5 \text{ C}_2 \text{ O}_4^{2-} + 16\text{H}^+ \rightarrow 2 \text{ Mn}^{2+} + 10\text{CO}_2 + 8\text{H}_2 \text{O}.$  (4+5+4=13)