Reg. No. □□□□□□□□□□

IV Semester M.Sc. Degree Examination, September/October 2022
(CBCS 2016 – 17 Syllabus)
ANALYTICAL CHEMISTRY
Optical Methods of Analysis

Time : 3 Hours   Max. Marks : 70

Note :   i)   Answer Part – **A** and **any four** questions from Part – **B**.
             ii) Figures to the **right** indicate **marks**.

PART – A

Answer **all** the following sub-divisions :   **(9×2=18)**

1. a) Explain why the radiation source in AAS is usually modulated.
   b) Enumerate the types of fuel and oxidants and their temperature ranges used in flame AAS.
   c) Why electrothermal atomizer is more sensitive than flame in AAS ? Explain.
   d) Indole exhibits maximum emission at the following wavelengths (nm) in different solvents upon excitation at 285 nm : 297 (cyclohexane), 305 (benzene), 310 (1, 4-dioxane), 330 (ethanol) and 350 (water). Explain this observation.
   e) What is the difference between a filter fluorometer and spectrofluorometer ?
   f) Why spectrofluorometry is more sensitive than spectrophotometry ?
   g) Explain the principle involved in photoelectron spectroscopy.
   h) Distinguish between nephelometry and turbidimetry.
   i) What is surface chemistry ? Explain.

PART – B

Answer **any four full** questions :   **(4×13=52)**

2. a) Explain the chemical interferences encountered in AAS.
   b) Explain the cold vapour AAS method for the determination of mercury.
   c) A serum sample is analysed for calcium by FES using the method of standard additions. Two 0.5 ml aliquots are added to 5.0 ml portions of water. To one portion is added 0.05 mL of 0.04 M $CaCl_2$ solution. The net emission signals in arbitrary units are 52.2 and 80.6 respectively. Calculate the concentration of calcium in the serum.   **(5+5+3)**

3. a) With a neat diagram, explain the working of a hollow cathode lamp.

   b) Discuss the procedure involved in the determination of sodium in water sample by flame photometry.

   c) Describe the working of an atomic absorption spectrophotometer.   **(4+4+5)**

4. a) Draw the Jablonski diagram and explain the various deactivation processes.

   b) Give the block diagram of a spectrofluorometer and explain its working.

   c) What is quantum efficiency ? Distinguish between fluorescence and phosphorescence.   **(5+5+3)**

5. a) Explain with examples, how the structural features and temperature of the medium affect the fluorescence intensity of molecules ?

   b) Discuss the fluorometric analysis of inorganic and organic compounds.

   c) Derive the equation that relates fluorescence intensity (F) to the concentration (c) of a fluorophore. Why is the linearity between F and c lost at high concentrations ?   **(5+5+3)**

6. a) Explain the effect of concentration, particle size and wavelength on scattering.

   b) Discuss how photoelectron spectroscopy is useful for structural elucidation.

   c) With a suitable example, discuss the use of turbidimetric titration for quantitative analysis.   **(4+5+4)**

7. a) Discuss the nephelometric determination of sulphate in a water sample.

   b) Describe how binding energy and oxidation state can be studied by photoelectron spectroscopy.

   c) A sample of barium chloride weighed 0.5 g. 50 ml of 0.2 N $AgNO_3$ solution was added to precipitate chloride of sample as AgCl and excess $AgNO_3$ was titrated with 0.28 N KSCN to give the titre value of 25.5 ml. Calculate the amount of chloride.   **(5+5+3)**

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