

This question paper contains 4 printed pages]

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S. No. of Question Paper : **5702**

Unique Paper Code : **2173012016**

Name of the Paper : **Analytical Methods in Chemical Analysis (DSE)**

Name of the Course : **B.Sc. (H) Chemistry**

Semester : **IV**

Duration : **3 Hours**

Maximum Marks : **90**

(Write your Roll No. on the top immediately on receipt of this question paper.)

Use of Scientific Calculator is not allowed.

Attempt any *six* questions.

Question No. 1 is compulsory.

1. Attempt any *five* of the following :

- (a) What are the purposes of the F-test and Q-test in statistical analysis of data ?
- (b) Define Spectroscopy. Discuss the origin of a spectra in spectroscopy.
- (c) How does the choice of flame and atomization method affect the sensitivity and accuracy of these techniques ?
- (d) Explain Nernst's distribution law and state its limitations.
- (e) Discuss the factors to be considered while selecting a chromatographic technique for separation of a sample.
- (f) How does a DTG curve enhance the interpretation of data obtained from a TGA experiment ?

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P.T.O.

2. (a) Define the term analytical chemistry. Define sampling. What are the key considerations for obtaining a representative sample ? How does the gross sample is different from grab sample ?
- (b) An enzymatic method for determining alcohol in wine is evaluated by comparison with a gas-chromatographic (GC) method. The same sample is analyzed several times by both methods with the following results (% ethanol). Enzymatic method : 13.1, 12.7, 12.6, 13.3, 13.3. GC method : 13.5, 13.3, 13.0, 12.9. Does the enzymatic method give the same value as the GC method at the 95% confidence level ? (Given that F-tabulated value at the 95% confidence level is 9.12 and t -tabulated value at the 95% confidence level is 2.365).
- (c) Define error. Differentiate between systematic errors and random errors. Suppose that in a gravimetric analysis, you forget to dry the filter crucibles before collecting precipitate. After filtering the product, you dry the product and crucible thoroughly before weighing them. Is the apparent mass of product always high or always low ? Is the error in mass systematic or random ? 5,5,5
3. (a) State Lambert-Beer's law. Give the mathematical expression for Beer-Lambert's law. How does a double beam spectrophotometer differ from a single beam spectrophotometer ?
- (b) A sample in a 1.0 cm cell is determined with a spectrophotometer to transmit 80% light at a certain wavelength. If the molar absorptivity of this substance at the wavelength is 4.0. What is the concentration of the substance ?

- (c) Describe the working principles of phototube and photomultiplier tube detectors used in UV-Visible spectrophotometry. Why is the photomultiplier tube generally preferred over the phototube in modern spectrophotometers ? 5,5,5
4. (a) Draw a well-labeled block diagram of Atomic Absorption Spectrophotometer (AAS) and explain the function of each part. Discuss the advantages and limitations of AAS technique.
- (b) Explain spectral, chemical and ionization interferences, which are commonly encountered in Flame Emission Spectroscopy (FES) with suitable examples. How can these interferences be minimized or corrected during analysis ?
- (c) Define background absorption in FES. Explain about Zeeman effect and Smith-Hieftje background correction methods. 5,5,5
5. (a) Multiple step Extraction is more efficient as compared to single step extraction. Comment.
- (b) Explain Continuous Extraction. What is the difference in Continuous and Batch extraction processes ?
- (c) In the solvent extraction process of uranium using 8-hydroxyquinoline in chloroform, both the aqueous and organic phases had volumes of 25 ml. If the percentage extraction was 99.8%, determine the distribution ratio. 5,5,5
6. (a) What is a thermogram ? Discuss the TGA curve of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$.
- (b) Discuss the TGA analysis for mixture of Calcium and Magnesium Carbonates where significant mass lost are observed at 200°C , 500°C

and one in between 600-900°C. How will you calculate the mass of CaO and MgO in the sample ?

- (c) What information can be obtained by Differential Thermal Analysis (DTA) that cannot be obtained by TGA ? 5,5,5

7. (a) A triplicate analysis of an ore in a laboratory by titration yields the following results : 92.50. 92.58. 92.42 wt.% of the pure compound. Within what range are you 95% confidence that the true value lies ? (At 95% confidence level and two degree of freedom, $t = 3.830$).

- (b) Explain the concept of charge transfer transitions in UV-Vis spectroscopy. How do these transitions occur, and in what types of compounds are they commonly observed ?

- (c) Distinguish between Chromophore and Auxochrome and give *two* examples each of Chromophore and Auxochrome. 5,5,5

8. (a) Explain Frontal, Elution and Displacement methods of Chromatography.

- (b) Describe thin layer chromatography (TLC) and highlight its differences from column chromatography.

- (c) Define resolution in chromatography and discuss factors that affect it. 5,5,5