

(2½ Hours)

[Total Marks : 75]

- N.B. :** (1) All questions are compulsory.
 (2) Figures to the right indicate full marks.
 (3) Use of log table / non-programmable calculator is allowed.

1. Answer any three of the following :-

15

- A) Explain 'Null hypothesis'.
 B) A volumetric analysis for calcium ions on triplicate samples of blood serum of a patient gave the following data :

Ca ²⁺ (mmol dm ⁻³)	3.15	3.25	3.26
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What is the 95% confidence interval for the mean of the data, assuming :

- a) no additional information about the precision of the method is known, and
 b) a large number of measurements have given $\sigma = 0.056$ mmol dm⁻³ of calcium ions [Given : $t = 4.30$, and $z = 1.96$ at 95% confidence level]

- C) Define sampling. Explain the sampling of immiscible liquids.
 D) The following table gives the dependence of 'y' on 'x'

x	1	2	3	4	5
y	2.3	4.8	7.0	7.6	9.2

Assuming a linear relationship between the variables, derive an equation of the type $y = mx + c$ by least square method.

- E) Describe random and non-random sampling.
 F) Discuss the sampling of compact solids.

2. Answer any three of the following :-

15

- A) Mention various steps involved in HPTLC. Write any two advantages and limitations of HPTLC.
 B) Discuss the following w.r.t. TLC
 i) Two dimensional development
 ii) Any two applications
 C) Name any two detectors used in HPLC. Draw a neat labelled diagram of any one of these detectors, mentioning any two advantages.
 D) Give any five applications of HPLC.
 E) Define R_f value. Explain the classification of chromatographic techniques based on phases involved, giving examples of each type.
 F) Explain the different methods used for locating the separated components in paper chromatography.

[TURN OVER]

3. Answer any three of the following :- **15**

- A) Draw a neat labelled diagram of AAS and give the function of each component.
- B) Discuss total consumption burner, mentioning its function, with a neat labelled diagram. State the factors which control the temperature of the flame of burner.
- C) Derive a mathematical relationship between intensity of fluorescent radiation and the concentration of the solution.
- D) Give any five applications of AAS.
- E) Draw a neat labelled diagram of a turbidimeter and explain turbidimetric calibration curve.
- F) Explain : i) turbidimetric titration, and
ii) phase titration

4. Answer any three of the following :- **15**

- A) 10.0 cm³ of 0.1 M ferrous ion solution is titrated with 0.1M ceric ion solution. Calculate the potential of the system at i) half the equivalence point, and (ii) the equivalence point.

$$[E^0_{\text{Pt/Fe}^{+3}, \text{Fe}^{+2}} = 0.770\text{V and } E^0_{\text{Pt/Ce}^{+4}, \text{Ce}^{+3}} = 1.44\text{V}]$$

- B) Draw a neat labelled diagram of a double beam spectrophotometer. Give the function of any two components.
- C) Explain the important analytical applications of UV-Visible spectroscopy.
- D) Explain the extraction of metal ions by chelation.
- E) What is the principle of Craig's counter current extraction. With the help of neat diagram, describe Craig's apparatus.
- F) Write an informative note on 'solid phase extraction'.

5. A) Fill in the blanks :- **4**

- a) The peak of normal error curve indicates error.
- b) In F test, 'F' is defined as the ratio of of two sets of observations.
- c) Gaussian distribution curve is a plot of relative frequency of error against magnitude of
- d) The sample prepared by mixing the increments is called sample.

OR**A) State true or false :-** **4**

- p) The value of Pearson's correlation coefficient, close to -1 indicates that the dependent variables and independent variables are not linearly related.
- q) In the least square method, the sum of squares of deviations of all the points should be maximum.

[TURN OVER]

- r) Sample containing boron is stored in borosilicate glass containers.
- s) For dissolution of acidic samples, basic fluxes are used.

5. B) Fill in the blanks :-

4

- a) In chromatographic technique, the components of the mixture are carried through the stationary phase by the flow of phase.
- b) If two or more solvents which differ in polarity, are employed in HPLC, it is known as elution.
- c) The role of precolumn is to remove from solvent to prevent contamination of the analytical column.
- d) In HPLC, separation is carried out at temperature.

OR**B) State true or false :-**

4

- p) HPTLC is a planar chromatographic technique.
- q) Screw driven syringe type pump, produces pulse free delivery in HPLC.
- r) Degassing system is employed in HPLC to remove dissolved gases from solvent.
- s) It is not quite easy to achieve reproducibility of R_f values in TLC.

5. C) Fill in the blanks :-

4

- a) Fraction of free atoms that are thermally excited is governed by distribution equation.
- b) In the expression, total amount of light absorbed at $\nu = \frac{\pi e^2}{mc} Nf$, 'f' stands for
- c) The phenomenon in which emission of light continues even after the exciting radiation is cut off, is called
- d) Liquid is used to cool the sample to enhance the phenomenon of phosphorescence.

OR**C) State true or false :-**

4

- p) AAS is independent of flame temperature.
- q) If two elements present in the sample have absorption at the same wavelength, analysis give erroneous results in AAS.
- r) Phosphorimetry is more sensitive than fluorimetry.
- s) Phosphoroscope can measure phosphorescence in the presence of fluorescence.

[TURN OVER]

5. D) Fill in the blanks :-**3**

- a) In redox titrations, colour of oxidised form is seen over reduced form, when

$$\frac{[In_{(ox)}]}{[In_{(red)}]} \geq \dots\dots\dots$$

- b) In the titrations of Fe (II) vs $K_2Cr_2O_7$, the indicator diphenylamine is irreversibly oxidised to
- c) In UV-Visible spectroscopy, the calibration curve is a plot of versus concentration.

OR**D) State 'True' or 'False' :-****3**

- p) The cavity and nature of the crown ether greatly influence the extraction process.
- q) Tributylphosphate is an example of solvating solvent in synergistic solvent extraction.
- r) A tungsten lamp can be used as a source in UV-Visible spectrophotometer, to obtain visible light.
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