[Time: 2½ Hours]   **[Marks:75]**

Please check whether you have got the right question paper.

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

Q.1 Answer any three of the following: **15**

A) Discuss the sampling of heterogeneous liquids and flowing liquids.

B) Two different methods were used for analysis of magnesium from a given sample.
The results obtained gave percentage of magnesium as follows.

Method I	5.94	6.01	5.98
Method II	5.65	5.70	5.61

The combined standard deviation is 0.03%.
Determine whether the two methods differ statistically or only numerically.
(Given t_{table}=2.78, for 95% probability level)

C) Following results were obtained in the determination of chromium in colorimetric measurement.

Conc. of Cr (ppm)	3.0	6.0	9.0	12.0
Absorbance	0.068	0.137	0.206	0.273

If sample containing chromium gave an absorbance of 0.196, obtain the amount of chromium in the sample, using least square method.

D) Discuss the sampling of gases by displacement method, with a neat labelled diagram.

E) Give a brief account of confidence limit and confidence interval.

F) Describe a method for rejection of doubtful values in a set of measurements.

Q.2 Answer any three of the following: **15**

A) Give the classification of chromatographic method based on the phases involved with one example of each type.

B) Explain the following w.r.t TLC: a) spotting of sample   b) development of chromatoplate.

C) Discuss the applications of paper chromatography.

D) Explain the functions of pump in HPLC. Name any two types of pumps in HPLC giving an advantage and a limitation of each type.

E) Draw a neat labelled diagram of UV detector used in HPLC, discuss its working and gave its advantages.

F) Write any five applications of HPTLC.

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

A) Discuss the principle of AAS.

B) Draw a schematic diagram of flame photometer and describe any three factors affecting intensity of emitted radiations.

C) Give any five applications of flame photometry.

D) Explain: a) turbidimetric titrations, and
b) phase titrations.

E) Derive a mathematical relationship between the intensity of fluorescent radiations and concentration of the solution.

F) Draw a schematic diagram of turbidimeter and explain turbidimetric calibration curve.

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

A) Draw a neat labelled diagram of double beam spectrophotometer and explain the function of its components.

B) What are redox indicators? Discuss the use of ferroin indicator in the redox titration of Fe^{2+} against Ce^{4+}.

C) Calculate the potential at the equivalence point in the titration of $10.0cm^3$ of 0.1M Fe(II) solution against 0.02M $KMnO_4$ solution at pH=1.
Given: $E^o_{pt/Fe^{3+},Fe^{2+}} = 0.771V$
$E^o_{pt/MnO4^-,Mn^{2+}} = 1.510V$

D) Discuss the principle of solid phase extraction. Give any two advantages of solid phase extraction over solvent extraction method.

E) Discuss principle and applications of Craig's counter current extraction.

F) Explain the role of complexing agent in solvent extraction.

Q.5 A) Fill in the blanks: **04**

a) The minimum sized packages in the consignment which the sample may represent is called _____.

b) For dissolution of silicate rock samples, _____ acid is mainly used.

c) The most common fluxes used for decomposition of sample are compounds of _____ metals.

d) Equation of Gaussian distribution curve is Y =_____.

OR

A) State true or false: **04**

p) Auger sampler is used for sampling of compact solids.

q) Sampling error is found to be directly proportional to the square root of number of samples averaged.

r) Coning & quartering method is used for the reduction of particle size.

s) Mixing of increments give sub-sample.

B) Fill in the blanks: **04**

a) In paper chromatography, _____ value is used to identify separated components.

b) Development of chromatogram requires_____time in TLC than paper chromatography.

c) The HPLC, if the composition of mobile phase is same then it is called as _____ elution.

d) Thickness of the stationary phase in HPTLC is _____ than that in TLC.

OR

B) State true or false: **04**

p) The mode of separation in case of paper chromatography is adsorption.

q) Separations by TLC are sharper than that of paper chromatography.

r) HPLC separations are carried out elevated temperature.

s) Post chromatographic derivatisation is possible in HPTLC.

C) Fill in the blanks: **04**

a) The process in which atoms of the element are dislodged from the surface of the cathode and transformed in to gaseous atoms, in hollow cathode lamp is called _____.

b) Phosphoroscope measures _____ in presence of fluorescence.

c) In AAS, the steady light from hollow cathode lamp is converted into pulsating light by _____.

d) Life time of phosphorescence is _____ than fluorescence.

OR

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C) State true or false: **04**
- p) Phosphorescence is not readily observed at room temperature.
- q) The choice of nephelometry and turbidemetry depends on the fraction of light scattered.
- r) Flame serves as a radiation source for different elements in AAS.
- s) If an atom of higher atomic number is introduced into an electron system, it enhances phosphorescence.

D) Fill in the blanks: **03**
- a) The phenomenon of extraction in which two reagents, when used together, extract a metal ion with enhanced efficiency compared to their individual action is called _____ extraction.
- b) Calibration curve, in UV-visible spectrophotometry is a plot of _____ against concentration of solution at a wavelength.
- c) In the redox titration of Fe(II) v/s $K_2Cr_2O_7$, diphenylamine is first oxidized to colourless substance known as _____.

OR

D) State true or false: **03**
- p) Grating are made up of non-corrosive materials.
- q) UV-visible spectra is normally used for detection of functional groups.
- r) One of the factor influencing extraction with crown ethers is size of the cation.

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