

T.Y. B.Sc. - chemistry - Paper IV  
Sub- Analytical chemistry  
Sem-V 2016-17

QP Code : 78951

(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 :-

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- A) Explain least square method for obtaining the slope of a plot passing through the origin.
- B) A chemist obtained the following data for percentage of lindane, in triplicate analysis, of an insecticide preparation :  
7.63, 7.48 and 7.53%  
Calculate the 90% confidence interval for the mean of the data assuming that  
i) no additional information about the precision of the method is known, and  
ii) a large number of measurements gave  $\sigma = 0.28\%$   
[Given :  $t = 2.92$ , and  $z = 1.64$  at 90% confidence level]
- C) Name the different methods used for sampling of stack gases and describe any one of these in brief.
- D) Define normal error curve with a neat diagram. A standard method for the determination of carbon monoxide level in gaseous mixtures is known to have standard deviation of 0.21 ppm. A modification of the method yields a value for standard deviation of 0.15 ppm. Verify, using F test, whether the two standard deviations are same or different. [Given  $F_{table} = 2.30$ ]
- E) Write a note on preservation and dissolution of sample.
- F) Name the different methods used for reduction of sample size in sampling of solid and discuss any one of these, in brief.

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2. Answer any three of the following :-

- A) Explain the following w.r.t. TLC.  
i) Preparation of plate  
ii) Ascending development.
- B) Give any five applications of HPTLC.  
Draw a neat labelled diagram of a typical HPLC unit and explain the following.  
i) Isocratic and gradient elution  
ii) Function of precolumn

[ TURN OVER ]



- D) What are the various steps involved in the technique of paper chromatography. Give any four applications.
- E) Explain the function of pump in HPLC. Name any two types of pumps used in HPLC giving one advantage and limitation of each type.
- F) Define  $R_f$  value. Give the classification of chromatographic techniques based on phases involved, giving suitable examples.

3. Answer any three of the following :-

- A) Draw a neat labelled diagram of a flame photometer and explain the function of each component.
- B) Discuss : i) Determination of molecular weight of macromolecules by the turbidimetric method.  
ii) Phase titration.
- C) Explain the principle of AAS.
- D) Draw a schematic diagram of single beam fluorimeter and explain its use in the determination of concentration of the sample solution.
- E) Give any five applications of flame photometry.
- F) Explain the factors affecting fluorescence.

4. Attempt any three of the following :-

- A)  $10.0\text{cm}^3$  of  $0.1\text{M}$  Fe (II) solution is titrated with  $0.1\text{M}$  Ce (IV) in acidic medium. Calculate the potential i) at half the equivalence point, and ii) on addition of  $11.1\text{cm}^3$  of titrant. [ $E^\circ_{\text{Pt/Fe}^{+3},\text{Fe}^{+2}} = 0.770\text{V}$  and  $E^\circ_{\text{Pt/Ce}^{+4},\text{Ce}^{+3}} = 1.44\text{V}$ ]
- B) What are redox titrations? How does diphenylamine behave as an indicator in redox titrations? What is the function of  $\text{H}_3\text{PO}_4$  in such titrations?
- C) What are the light dispersing devices used in UV-Visible spectrophotometers? Describe any one of these devices, in brief.
- D) Write a note on synergistic solvent extraction.
- E) Describe Craig's counter current extraction.
- F) Explain the principle of solid phase extraction. Give any three advantages of solid phase extraction over solvent extraction methods.

5.A) Fill in the blanks :-

- a) When all the observations recorded are with equal frequency without any prejudice, then the value of chi-square is .....
- b) In Q-test,  $Q_{\text{cal}}$  is obtained by dividing the difference between the doubtful measurement and its neighbouring measurement by ..... of the set.



- c) When the two variables x and y are not linearly related, the value of correlation coefficient is .....
- d) A stated amount of material to be taken from sampling unit is called .....

OR

- A) State true or false :-
- p) Split-barrel sampler is used to collect samples of compact solids.
- q) The peak of normal error curve indicates maximum error.
- r) The test used by statisticians to determine relationship between dependent variables and independent variables is Null hypothesis.
- s) Mixing of increments gives a sub-sample.

## 5.B) Fill in the blanks :-

- a) In paper chromatography ..... value is used to identify separated components.
- b) Densitometer is used as a ..... in HPTLC.
- c) In HPLC, separation is carried out at ..... temperature.
- d) Thickness of stationary phase in HPTLC is ..... than that in TLC

OR

## B) State true or false :-

- p) TLC separation is based on adsorption.
- q) Refractive index detectors are highly temperature sensitive.
- r) In HPLC columns are reusable.
- s) UV - visible detector in HPLC is based on absorption of UV or visible radiation.

## 5.C) Fill in the blanks :-

- a) An expression of Boltzman distribution equation is  $\frac{N^x}{N_0} = \dots\dots\dots$
- b) In AAS, the steady light from hollow cathode lamp is converted into pulsating light by .....
- c) Phosphorimetric experiments are normally carried out at ..... temperature.
- d) In nephelometry, the detector is usually, but not necessarily, placed at ..... angle to the incident radiation.

OR

## C) State true or false :-

- p) Electrothermal atomiser is also called graphite furnace.
- q) Flame serves as a radiation source for different elements in AAS.
- r) Fluorescence is a delayed phosphorescence.
- s) In nephelometry, highly monochromatic radiation is not necessary.

[ TURN OVER ]



- D) Fill in the blanks :-
- An ideal redox indicator should exhibit a detectable colour change when the titrant causes a shift of potential of about ..... V.
  - In spectrophotometry, ..... lamp is used as a source of radiation for obtaining UV light.
  - In 12-crown-4, the number 4 indicates the number of ..... atoms.

OR

- D) State 'True' or 'False' :-
- For solvents with high dielectric constant, ion pair formation decreases with temperature.
  - UV - Visible spectroscopy is used for the detection of functional groups.
  - Chromophores are unsaturated groups which are responsible for the absorption of visible light.