

(Time: 3 hours)

Total Marks: 100

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.

Q.1

Attempt any four of the following.

20

- A) Define quality. Explain the terms: 1) quality control and 2) quality assurance.
- B) Determine the mass percent composition of each element in calcium nitrate. $\text{Ca}(\text{NO}_3)_2$
(Atomic weight of Ca = 40, N = 14, O = 16)
- C) Calculate molarity and molality of solution containing 8.5 g of NaCl dissolved in 0.7 dm³ of solution. Density of the solution is 1.0 gcm⁻³ (Given: atomic weight of Na = 23 and Cl = 35.5)
- D) What are difficulties encountered in sampling of solids? Explain the terms bulk ratio and size to weight ratio.
- E) Explain the importance of quality concepts in industry.
- F) Describe sampling of homogeneous and heterogeneous liquids.

Q.2

Attempt any four of the following.

20

- A) Discuss the theory of redox indicators and explain how ferroin indicator is suitable in the redox titration of Fe²⁺ versus Ce⁴⁺.
- B) What are metal ion indicators? Give the three properties of good metal ion indicator with suitable examples.
- C) What are complexometric titrations? Explain Back titration and Displacement titration.
- D) Derive an expression for potential of the system at the equivalence point and after the equivalence point in the titration of Fe (II) with Ce(IV) solution.
- E) What are complexometric titrations? Discuss any two techniques used to increase the selectivity of EDTA.
- F) 25.0 cm³ 0.1 M Fe (II) solution is titrated with 0.1M Ce (IV) in acidic medium Calculate the potential
i) at the equivalence point
ii) on addition of 12.5 cm³ 0.1 M Ce (IV)

Given : $E^0_{\text{Fe}^{3+}/\text{Fe}^{2+}} = 0.771 \text{ V}$
 $E^0_{\text{Ce}^{4+}/\text{Ce}^{3+}} = 1.44 \text{ V}$

Q.3.

Attempt any four of the following.

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- A) What are the different types of burners used in flame photometry? Explain any one of them.
- B) Explain electrothermal atomiser. Give any three limitations of AAS.
- C) Explain the principles underlying the emission of fluorescent light. How does fluorescence differ from phosphorescence?
- D) Explain phosphorescence. Give two applications of phosphorimetry.

- E) Draw a schematic diagram of turbidimeter and explain turbidimetric titrations using turbidimetric titration curve.
 F) Discuss the instrumentation of nephelometry with the help a labelled diagram.

Q.4

Attempt any four of the following.

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- A) What are the factors affecting solvent extraction? Explain any one.
 B) Discuss the principle of solid phase extraction. Give any two points of comparison between solvent extraction and solid phase extraction.
 C) What are the requirements for high pressure pump used in HPLC? Name any two high pressure pumps.
 D) Explain the role of precolumn used in HPLC. Give any two applications of HPLC.
 E) Name the detectors used in HPTLC and explain any one of these in brief.
 F) What are the advantages and limitations of HPTLC.

Q.5

A) Select the correct option and complete the following statements: (any five)

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- a) _____ materials cannot be used for validation of a method.
 i) Reference ii) Certified reference
 iii) Standard reference
 b) A chemical of lowest purity is _____ grade.
 i) GR ii) LR iii) AR
 c) The normality of 0.5 M H_2SO_4 is _____.
 i) 0.05 ii) 1.0 iii) 0.25
 d) 10 μ g of solute in 1 litre of solution is 10 _____ solution.
 i) ppm ii) ppb iii) ppt
 e) If solution contains 40 g of KCl and 60 g of water, then it contains _____ % (w/w) KCl.
 i) 60 ii) 40 iii) 100
 f) _____ is used for sampling of compact solid.
 i) Split tube thief ii) concentric tube thief iii) split-barrel sampler
 g) _____ method is mostly used to draw sample from a cylinder filled with gas at higher temperature.
 i) Flushing ii) Displacement iii) Expansion
 h) _____ is excellent solvent used for dissolution of many metal oxides.
 i) Conc. HCl ii) Conc. H_2SO_4 iii) Conc. HNO_3

Q.5

B) State whether true or false: (any five)

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- a) Murexide is used as indicator in Fe^{2+} versus Ce^{4+} titration.
 b) Ferroin indicator is a complex of 1-10 phenanthroline with Fe (II)
 c) PO_4^{3-} can be titrated by direct titration of EDTA
 d) EDTA is standardised by using standard solution of $ZnSO_4$
 e) During titration of Fe (II) with Ce (IV) the potential after equivalence point depends on ratio of $[Fe^{2+}] / [Fe^{3+}]$

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- f) The transition potential of ferroin indicator is 0.76 V in 1 M H_2SO_4
- g) Eriochrome black T indicator exhibits blue colour between pH 7 to 11

Q.5 C)

Fill in the blanks: (any five)

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- a) _____ is the study of the emission of radiation by element when their salt solutions are sprayed into the flame.
- b) Only small portion of sample reaches the flame in _____ burner of flame photometry.
- c) In AAS, the steady light from hollow cathode lamp is converted into pulsating light by _____.
- d) Phosphorimetric experiments are normally carried out at _____ temperature.
- e) Secondary filter in fluorimetry absorbs _____ light.
- f) In _____ the detector is mounted on a turntable for positioning at different angle.
- g) A turbidimeter measures _____ as a function of concentration of suspensions.
- h) A nephelometer measures the intensity of _____ light.

Q.5 D)

Match the columns: (any five)

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- | Column A | Column B |
|------------------------------------|--|
| a) Difference in $pH_{1/2}$ values | (i) Sample application as bands or spots |
| b) HPTLC | (ii) Measure of separability of two ions |
| c) Refractive index detector | (iii) Applicable to solute exists in same molecular form |
| d) UV-Detector | (iv) Extraction of Fe (II) in ether in acidic medium |
| e) Partition coefficient | (v) Highly temperature sensitive detector |
| f) Ion pair formation | (vi) Multistage separation |
| g) Counter current extraction | (vii) Mercury source with 254 and 280 nm wavelength |
