[3 Hours]

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 log table/non-programmable calculator is allowed.
- 1. Answer any four of the following.

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[Total Marks: 100]

- A) Explain the importance of quality concepts in industry.
- B) Arsenic can be determined by titration with iodine solution. The reaction involved is  $HAsO_4 + I_2 + 2H_2O \rightarrow H_3ASO_4 + 2H^+ + 2I^-$ . A sample of pure  $As_2O_3$  weighing 4.0136 g is dissolved in water to make 800 cm<sup>3</sup> of solution. Calculate normality of  $As_2O_3$  solution used for standardizing iodine solution and convert it into its molarity. [Given: molecular weight of  $As_2O_3 = 197.84$ ].
- C) What is the normality of 69% (w/w) HNO<sub>3</sub>, having a density of 1.42 g cm<sup>-3</sup>. Using this HNO<sub>3</sub>, (69%), how would you prepare 250 cm<sup>3</sup> 2N HNO<sub>3</sub>?[Given: molecular weight of HNO<sub>3</sub>=63]
- D) Discuss the sampling of static liquids.
- E) Discuss any one method of sampling of stack gases, with a labelled diagram.
- F) Explain the importance of chemical standards and certified reference materials in chemical analysis.
- 2. A) What are metal ion indicators? Discuss its theory and give one example.

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- B) Name the different methods to increase the selectivity of EDTA. Discuss any two of these methods in brief.
- C) Calculate the potential at the equivalence point in the titration of 20.0 cm<sup>3</sup> of 0.1M Fe(II) solution against 0.02M KMnO<sub>4</sub> solution at pH=2.

Given:  $E^0_{\text{pt/Fe}}^{3+}, Fe^{2+} = 0.771V$  $E^0_{\text{pt/MnO}^4}, Mn^{2+} = 1.510V$ 

- D) 10.0cm<sup>3</sup> of 0.1M Fe(II) is titrated with 0.1M Ce(IV) in acidic medium. Calculate the potential on addition of
  - i) 1.0cm<sup>3</sup> of titrant, and
  - ii) 10.1 cm<sup>3</sup> of titrant

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

- E) Discuss the use of diphenylamine indicator in the redox titration, explaining the reactions involved and the function of H<sub>3</sub>PO<sub>4</sub> in such titrations
- F) Give any five advantages of EDTA, as a titrant.
- 3. Answer any four of the following:

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- A) Discuss the principle of FES.
- B) Distinguish between the following:
  - a) Fluorimetry and Phosphorimetry
  - b) Fluorimetry and Absorptiometry
- C) Give any five applications of AAS.
- D) Describe the construction and working of graphite furnace with a labelled diagram.
- E) Discuss the applications of turbidimetry in:
  - a) the determination of molecular weight of macromolecules
  - b) phase titrations.

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F) Draw a neat labelled diagram of nephelometer and explain its working. **4.** Answer **any four** of the following 20 A) Explain Craig's counter current extraction with a neat diagram, giving any two applications. B) Discuss the principle and process with reference to solid phase extraction. C) Draw a graph of percentage extraction versus pH and explain the concept of [pH]<sub>1/2</sub> and its significance. D) Draw a neat labelled diagram of a typical HPLC unit and mention the function of any three important components. E) Give any five applications of HPLC. F) Name the detectors used in HPTLC and discuss any one of these in brief. **5.** A) Answer **any five** of the following in one sentence. 05 a) What are laboratory reagents? b) What is the percentage composition of 'Na' in sodium hydroxide? (Na = 23, O = 16, H = 1)c) A solution contains 1mol of sugar and 19 mol of water. What is the mole of fraction of d) Give a mathematical expression for size to weight ratio, 'b', with reference to sampling e) Name any one device to be used for sampling of particulate solids. f) Name the equipment used for sampling of flowing liquid. g) What is the colour change at the end point in the redox titration of Fe(II) versus Ce(IV) in the presence of ferroin indicator? h) What is the role of coning and quartering method w.r.t. sampling of solids? **5.** B) Answer **any five** of the following in one sentence. 05 a) What is the criteria for selection of indicator in redox titrations? b) What are redox indicators? c) What should be the ratio,  $\frac{[In_{(0x)}]}{[In_{(red)}]}$  in order to see the colour of oxidized form of an indicator in the redox titrations? d) What is tris(1,10-phenanthroline)iron (II) sulphate commonly known as? e) Give the general equation representing the formation of metal-EDTA complex. f) What is the role of auxilliary complexing agent in direct titrations w.r.t. EDTA titrations? g) Name any one most commonly used standard metal ion solution in back titration. h) Give an example of indirect titration with respect to EDTA titrations. C) Fill in the blanks: (any five) 05 a) Phosphoroscope measures in the presence of fluorescence. b) Phosphorimetric experiments are normally carried out at \_\_\_\_\_ temperature. c) A process in which atoms of the element are dislodged from the surface of the cathode and transformed in the gaseous atoms, in hollow cathode lamp is called \_ d) If an atom of higher atomic number is introduced in to a  $\pi$ -electron system, it the fluorescence.

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e) The function of secondary filter, in fluorimeter, is to absorb \_\_\_\_\_ radiations.

- f) In AAS, the steady light from hollow cathode lamp is converted in to pulsating current by \_\_\_\_\_.
- g) Only a small portion of sample reaches the flame in \_\_\_\_\_ burner in flame photometry.

## (D) State true or False

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- a) HPLC separations are carried out at elevated temperature.
- b) Quantity of sample required in HPTLC is more than that in TLC.
- c) HPTLC can simultaneously handle several samples of divergent nature and composition, at given time.
- d) Screw driven syringe type of pump produces pulse free delivery in HPLC.
- e) Thickness of stationary phase in HPTLC is less than that in TLC.
- f) In HPLC, if the composition of the mobile phase changes with time, it is called as isocratic elution.
- g) Only volatile components can be analysed by HPLC.



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