2018

CHEMISTRY

CHM 302

ANALYTICAL TECHNIQUES

Full Marks: 80

Time: 3 hours.

The figures in the margin indicate full marks for the questions

1. **Answer the following questions (any four) :**   5X4=20

   (a) What is reverse phase HPLC? What is 'Gradient' condition HPLC analysis? State its advantages over 'Isocratic' condition? What is preparative HPLC?

   (b) What are ion-exchange resins? Discuss briefly the principle of ion-exchange chromatography.

   (c) Discuss the principle of HPTLC and its advantages over TLC.

   (d) Discuss the principle of GC-MS. What are the advantages of Flash chromatography on TLC?

   (e) What are the theoretical plates? Discuss any one theory of chromatography.

   (f) Explain the mechanism of a continuous eluent suppression system used in ion chromatography, with a suitable diagram. How is pH useful in solvent extraction method?

2. **Answer the following questions (any two) :**   5X2=10

   (a) Explain with reasons the various precautions that should be taken before and during a cyclic voltammetry experiment.   5

(b) What do you mean by EC and ECE mechanisms of electrochemical reactions? How can these two mechanisms be distinguished by cyclic voltammetry? $2+3=5$

(c) Taking ferrocene as an example state how cathodic and anodic peaks are obtained in CV experiment. 5

3. **Answer the following questions (any four):** $5 \times 4 = 20$

(a) How can DSC be helpful in the construction of a phase diagram for a binary eutectic system?

(b) The following table summarizes some data about three Fe(III) chlorides. Sketch the TG curve anticipated if 25.0 mg sample of $FeCl_3.6H_2O$ is heated from 0°-400°C. 5

Compound	Molecular weight	Melting point (0°C)
$FeCl_3.6H_2O$	270	37
$FeCl_3.5/2H_2O$	207	56
$FeCl_3$	165	306

(c) In the TGA of 0.250g of $Ca(OH)_2$, the loss in weight at different temperatures was:

(i) 0.018g at 100-150°C (loss of hydroscopic water)

(ii) 0.038g at 500-560°C (dehydration)

(iii) 0.0229 at 900-950°C (dissociation)

Determine the composition of calcium hydroxide.

(d) What do you mean by structure factor? How is it related to intensity and atom form factor? $2+3=5$

(e) Derive the condition of systematic absence for FCC crystal lattice. 5

4. **Answer the following questions (any two):** $2 \times 5 = 10$

(a) What kind of information can be extracted from SEM and TEM? Write the working principle of TEM. $3+2=5$

(b) How many ways a AFM can be operated? Discuss about the advantages and disadvantages of AFM. $1+2+2=5$

(c) To record surface morphology of a rubber, which techniques will you prefer from the three techniques AFM, SEM and TEM and why? $1+4=5$

5. **Answer the following questions (any four):** $5 \times 4 = 20$

(a) Give at least one method of sample preparation for determining trace elements in the leaves of a plant. What precautions are necessary? $3+2=5$

(b) How does inductively coupled plasma mass emission spectroscopy work? Is it possible to determine all the elements with it? 5

(c) How can you determine trace amount of phenol in water? Give the complete method. 5

(d) You have to sample air for determining the presence of Pb and PAH in it. Describe how you will proceed. 5

(e) Anodic stripping voltammetry can accurately determine many trace elements in water. Explain the working principle of inductively coupled plasma torch with a suitable diagram. 5

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