MODERN INSTRUMENTAL METHODS OF ANALYSIS
(CHEN 4131)

Time Allotted : 3 hrs **Full Marks : 70**

Figures out of the right margin indicate full marks.

Candidates are required to answer Group A and any 5 (five) from Group B to E, taking at least one from each group.

Candidates are required to give answer in their own words as far as practicable.

Group – A
(Multiple Choice Type Questions)

1. Choose the correct alternative for the following: **10 × 1 = 10**

(i) Select the wavelength range corresponding to UV-visible region.
(a) 400-800 nm (b) 200-800 nm (c) 25 μm-2.5 μm (d) 2.5 μm – 1 mm

(ii) The most widely used wavenumber range in IR spectroscopy is
(a) 15000 cm^{-1}-12000 cm^{-1} (b) 4000 cm^{-1}-670 cm^{-1}
(c) 400 cm^{-1}-200 cm^{-1} (d) 6000 cm^{-1}-5000 cm^{-1}

(iii) X-ray diffractometers are not used to identify the physical properties of which of the following?
(a) Metals (b) Liquids (c) Polymeric materials (d) Solids.

(iv) The possible transitions for water molecule in UV-visible region are
(a) σ →σ * (b) n→π *, π→π * (c) σ→σ *, n→π * (d) n → σ*

(v) In a gas chromatography with capillary column, phase ratio is given as
(a) Ratio of column radius to twice the stationary phase film thickness
(b) Ratio of twice the stationary phase film thickness to column radius
(c) Ratio of column radius to stationary phase film thickness
(d) Ratio of stationary phase film thickness to column diameter.

(vi) Consider a plane intersecting X-axis at a and runs parallel to Y and Z axes. The Miller index of the plane is
(a) (010) (b) (100) (c) (001) (d) (101)

(vii) In infrared spectroscopy, irradiation of a molecule with infrared light triggers
(a) Molecular translational motion (b) Molecular vibrations
(c) Molecular rotation (d) Bond breaking.

(viii) Kα line emission from a target results due to electron transition from
(a) L shell to K shell (b) M shell to K shell (c) M shell to L shell (d) L shell to M shell.

(ix) Example of spectral interference in atomic emission spectroscopy is
(a) chemical interference (b) excitation interference
(c) ionization interference (d) background radiation.

(x) Organic Nitrogen compounds are detected using
(a) flame photometric detector (b) flame thermionic detector
(c) electron capture detector (d) sulphur chemiluminescence detector.

Group- B

2. (a) Define the principle of AAS analysis. Indicate whether analysis by AAS is quantitative or qualitative. What types of sample can be analyzed by AAS? [(CO1,CO2)(Apply/HOCQ)]
(b) Describe the difference between flame photometry and AAS. [(CO1,CO2)(Remember/LOCQ)]
(c) Describe the role of monochromator in AAS. [(CO1,CO2)(Remember/LOCQ)]
(3 + 2 + 1) + 3 + 3 = 12

3. (a) Describe the working principle of UV-Vis spectrophotometer with a labelled schematic diagram. [(CO2)(Understand/IOCQ)]
(b) Describe the strength and limitation of UV-Vis analysis. [(CO2)(Understand/IOCQ)]
6 + 6 = 12

Group - C

4. (a) "Intensity of incident radiation on a photomultiplier tube is measured by the number of electrons emitted in the tube" Justify the statement with discussion of the working principle of photomultiplier tube with a diagram. [(CO2)(Analyze/HOCQ)]
(b) The outer shell electrons can only be excited in Atomic Absorption Spectrometry. Comment on the validity of the statement through explanation of the basic principle of AAS. [(CO2)(Analyze/IOCQ)]
(c) "Hollow cathode lamp can overcome the problems associated with conventional radiation sources in AAS". Comment on the validity of the statement. [(CO1)(Remember/LOCQ)]
4 + 4 + 4 = 12

5. (a) "Atomic absorption and emission linewidths should ideally be narrow". Justify the statement with proper explanation of the different broadening effects on spectral lines. [(CO2)(Apply/IOCQ)]
(b) "A 25 K increase in flame temperature results in a 8% increase in the excited state population of potassium atoms that give rise to emission lines". Justify the statement. [(CO2)(Evaluate/HOCQ)]
(c) Distinguish between arc and spark type electrical excitation sources in emission spectroscopy. Sketch the current time profile in both excitation modes. [(CO2)(Understand/LOCQ)]
(4 + 1) + 4 + (1 + 2) = 12

Group – D

6. (a) State the application of "Fourier transform" in FTIR. Discuss the different sample handling techniques in FTIR. [(CO2,CO3)(Analyze/IOCQ)]

(b) Explain the basic difference in the working principle of EDXRF and WDXRF. State the applications of quantitative XRF. Write the equation relating concentration of an element with intensity of the signal detected in an XRF. [(CO2)(Understand/IOCQ)]

(1 + 5) + (2 + 3 + 1) = 12

7. (a) For the planes marked with arrows shown in Fig.1, determine the Miller indices and state how this information is useful in interpreting an X-ray diffractogram. [(CO1)(Apply/IOCQ)]

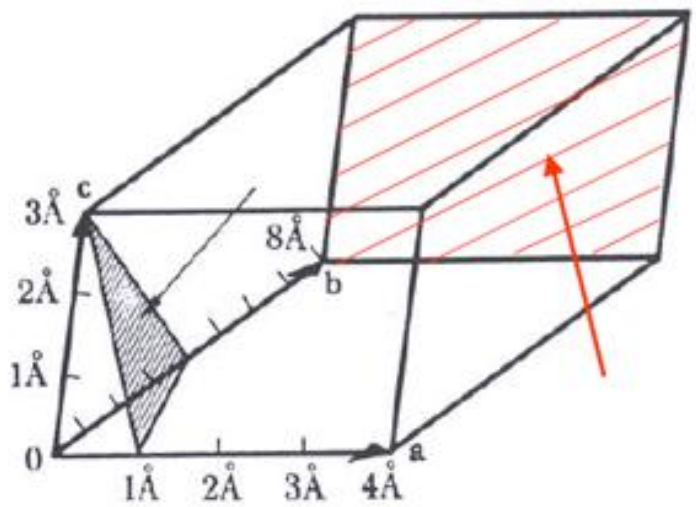

Fig. 1: Schematic of atomic plane

(b) Describe the sample preparation process for the XRD analysis. [(CO2)(Understanding/HOCQ)]

(c) In the X-ray diffraction of a set of crystal planes having d equal to 0.18 nm, first order reflection is found to be at an angle of 22°. Calculate the wavelength of X-rays. [(CO3)(Remember/LOCQ)]

4 + 4 + 4 = 12

Group - E

8. (a) A chromatogram with a single retained component is shown in the Fig.2 below. What information can be extracted from the chromatogram? [(CO2)(Remember/LOCQ)]

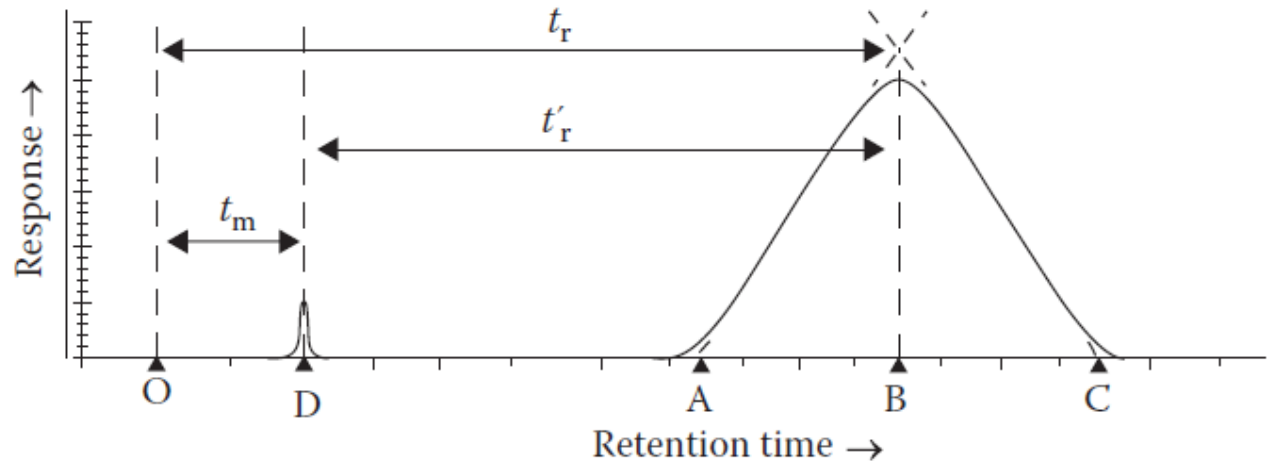

Fig.2: Chromatogram of a single component

(b) Define the following terms for a chromatographic separation process with their significances.
(i) Distribution ratio
(ii) Retention factor
(iii) Peak resolution
(iv) Relative retention factor. [(CO2)(Remember/LOCQ)]

(c) Discuss the criteria for selecting stationary and mobile phases in reverse phase HPLC. [(CO2,CO3)(Understand/LOCQ)]

3 + (4 + 2) + 3 = 12

9. (a) For a capillary GC column, 25 m long, with a diameter of 0.25 mm, operated at a constant temperature of 200°C under a helium pressure of 20 psi above atmospheric pressure, resulting in an average linear flow rate of 30.4 cm/s, the following data is given.

Peak	Retention time	Peak area				
1	3.70	64871	138589	300767	654611	1132301
2	4.00	53422	150838	345670	647345	1319829
3	4.10	65668	151856	235309	530813	1094460
4	4.30	54705	116870	253633	552025	954854
5	4.35	49904	140906	322911	604723	1232931
IS	8.00	287296	262670	270879	300977	279087
Concentration (ppm)		10	25	50	100	200

The typical number of theoretical plates were calculated as 30000.

(i) Calculate the HETP and predict about the column efficiency.

(ii) Calculate the relative retention times of the peaks.

(iii) Calculate the concentration of an unknown sample with peak area 194500 and retention time 4.12 min. [(CO2)(Evaluate/HOCQ)]

(b) Explain the principle of SPME.

8 + 4 = 12

Cognition Level	LOCQ	IOCQ	HOCQ
Percentage distribution	28.12	44.79	27.08

Course Outcome (CO):

After the completion of the course students will be able to

CO1: Define a problem where the determination and/or measurement of some chemical species is required.

CO2: Develop sufficient knowledge about the major instrumental methods of chemical analysis so that they can determine what technique should be used to solve a particular problem.

CO3: Perform and advice as expert the details of instrumental analysis techniques needed by industries solve problems of related field.

CO4: Gain the background necessary for a scientific expert witness to present new techniques in a court of law.

*LOCQ: Lower Order Cognitive Question; IOCQ: Intermediate Order Cognitive Question; HOCQ: Higher Order Cognitive Question.