

**MODERN INSTRUMENTAL METHODS OF ANALYSIS  
(CHEN 4131)**

**Time Allotted : 2½ hrs**

**Full Marks : 60**

*Figures out of the right margin indicate full marks.*

*Candidates are required to answer Group A and  
any 4 (four) from Group B to E, taking one from each group.*

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

**Group – A**

1. Answer any twelve:

**12 × 1 = 12**

*Choose the correct alternative for the following*

- (i) Which of the following analytical method is used to measure the analyte concentration depending on the quantity of light received by the analyte?  
(a) Spectroscopy (b) Decantation  
(c) Potentiometry (d) Specific surface area.
- (ii) The compound used to calibrate absorbance scale of the UV-visible instrument is  
(a) HCl (b) K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> (c) KCl (d) KMnO<sub>4</sub>.
- (iii) In atomic absorption spectroscopy the most strongly absorbed light is called as  
(a) Resonance line (b) Base line  
(c) Stokes line (d) Anti stokes line.
- (iv) The essential components of a NMR spectrometer are  
(a) Radiofrequency oscillator and superconducting magnet  
(b) UV radiation source and superconducting magnet  
(c) Infrared radiation source and superconducting magnet  
(d) Visible light source and superconducting magnet.
- (v) Evaporative light scattering detector is used in  
(a) GC (b) XRD (c) AAS (d) HPLC.
- (vi) In GC, all organic compounds are well detected using  
(a) ELCD (b) SP-FPD (c) NPD (d) FID.
- (vii) Selectivity of the stationary phase in chromatography is  
(a) Ratio of peak height of two components  
(b) Ratio of peak widths of two components  
(c) Ratio of partition constants of two components  
(d) Product of partition constants of two components.

- (viii) Stationary phase in HPLC is composed of  
 (a) n-C18 hydrocarbon (b) polymer  
 (c) metal (d) ceramic.
- (ix) Miller indices of a crystal plane intersecting the X axis at 1 and parallel to Y and Z axes are  
 (a) (100) (b) (010) (c) (110) (d) (001).
- (x) Detector used in arc and spark type atomic emission spectrometer is  
 (a) Thermocouple (b) Photomultiplier tube  
 (c) Golay detector (d) RI detector.

*Fill in the blanks with the correct word*

- (xi) The expression for Bragg's law is\_\_\_\_\_.
- (xii) XPS is used to determine\_\_\_\_\_.
- (xiii) AFS can be used to determine\_\_\_\_\_.
- (xiv) The photon of wavelength 400 nm corresponds to \_\_\_\_\_ wave number.
- (xv) Positive deviation from Beer's law is due to \_\_\_\_\_.

### Group - B

2. (a) State the Beer-Lamberts law. Describe the working principle of absorption spectroscopy with a neat schematic diagram. [[CO1](Analyse/HOCQ)]
- (b) Describe the difference between flame photometry and absorption spectroscopy. [[CO1](Apply/IOCQ)]
- (2 + 6) + 4 = 12**
3. (a) What are the limitations of instrumental analysis methods? [[CO1](Understand/LOCQ)]
- (b) State the various applications of absorption spectroscopy. [[CO1](Understand/LOCQ)]
- (c) Describe the properties of light required for the UV-Vis spectroscopy analysis. [[CO4](Remember/LOCQ)]
- 4 + 4 + 4 = 12**

### Group - C

4. (a) Discuss the working principle of a flame atomizer. [[CO1,CO2](Remember/LOCQ)]
- (b) Explain the advantages and disadvantages of using a hollow cathode lamp as radiation source by describing its working principle with a diagram. [[CO1,CO2](Understand/IOCQ)]
- (c) Discuss ways to eliminate interferences in AAS. [[CO2](Analyze/IOCQ)]
- 4 + 4 + 4 = 12**
5. (a) Distinguish between the two electrical excitation sources used in atomic emission spectroscopy. [[CO2,CO3](Analyze/IOCQ)]
- (b) Discuss the working principle of an AFS. State the applications of AFS. [[CO2](Understand/LOCQ)]

- (c) Discuss the significance of RU lines in sample analysis using arc and spark spectrometer. [[CO1,CO2](Analyze/IOCQ)]
- (d) Illustrate the different types of atomic fluorescence transitions. [[CO2](Apply/HOCQ)]
- 4 + 4 + 2 + 2 = 12**

### Group - D

6. (a) Distinguish between X-ray fluorescence spectroscopy (XRF) and X-ray photoelectron spectroscopy. [[CO1,CO2](Analyze/IOCQ)]
- (b) “The frequency of signal at the FTIR detector is governed by the relative velocity of the mirrors and the wavelength of radiation”. Justify the statement by explaining the working principle of an interferometer with a diagram. [[CO2,CO3](Analyze/HOCQ)]
- (c) Explain the different vibrational modes observed in covalent bonds upon infrared radiation with diagram. [[CO3](Remember/LOCQ)]
- 3 + 5 + 4 = 12**
7. (a) In the X-ray diffraction of a set of crystal planes having d equal to 0.23 nm, first order reflection is found to be at an angle of 25°. Calculate the wavelength of X-rays. [[CO3](Remember/LOCQ)]
- (b) Define full width half maxima with net schematic diagram. [[CO3](Remember/LOCQ)]
- (c) State the advantages and disadvantages of the electron microprobe method. [[CO3](Analyze/HOCQ)]
- 4 + 4 + 4 = 12**

### Group - E

8. (a) Explain the effect of increasing the column temperature and gas flowrate on gas and liquid chromatography. [[CO3,CO4](Analyze/HOCQ)]
- (b) Explain the criteria for selecting stationary and mobile phases for gas chromatography column. [[CO2,CO3](Understand/LOCQ)]
- (c) What form of chromatography is SPE? Distinguish between the working principle of SPE and SPME. [[CO4](Remember/LOCQ)]
- 4 + 4 + 4 = 12**
9. (a) Distinguish between normal and reversed phase chromatography. [[CO3,CO4](Apply/IOCQ)]
- (b) Distinguish between support particles of HPLC and packed column GC. [[CO2,CO3](Analyze/IOCQ)]
- (c) Explain the working of an HPLC pump. [[CO3,CO4](Remember/LOCQ)]
- (d) Distinguish between eluent and effluent. [[CO2](Apply/IOCQ)]
- 3 + 4 + 3 + 2 = 12**

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Cognition Level	LOCQ	IOCQ	HOCQ
Percentage distribution	44.79	31.25	23.96

**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.*