

**ANALYTICAL INSTRUMENTATION**  
**(AEIE 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) For a given column operated at temperature at T and carrier gas flow rate F, the length of time that an uncertain component spends in the gas chromatograph column is called  
(a) Dead time (b) Retention time  
(c) Retarded time (d) Delay Time
- (ii) Force acting on electron charge  $e$ , mass  $m$  moving with a velocity  $v$  in a direction parallel to uniform magnetic field  $H$  is  
(a)  $mv^2/r$  (b)  $Hev/r^2$   
(c) Zero (d) None of these
- (iii) The resistance of a glass electrode in a pH meter may be as high as  
(a)  $10^8 \Omega$  (b)  $10^5 \Omega$   
(c)  $10^{15} \Omega$  (d) None of these
- (iv) In NMR spectroscopy source used for interaction with sample is  
(a) Hollow cathode lamp (b) Spark generator  
(c) Radio frequency (d) None of these
- (v) Absorptivity is a property of a substance for a given wavelength will vary with  
(a) Concentration  
(b) Concentration and viscosity  
(c) Length of the light path  
(d) Concentration and length of the light path
- (vi) The conductivity of an electrolyte is determined by  
(a) Number and species of ions and their motilities  
(b) Number of ions only  
(c) Concentration of the solute  
(d) Temperature of measurement

- (vii) X-ray diffraction grating type monochromator obey the equation of  
(a) Beer's (b) Bragg's (c) Michelson's (d) None of these
- (viii) Concentration polarization of an electrode in electrochemical cell may be due to  
(a) Diffusion (b) Migration (c) Convection (d) All of these
- (ix) The cell that produces electrical energy is called  
(a) Electrolytic cell (b) galvanic cells  
(c) Reference cell (d) none of these
- (x) Physical property which distinguishes oxygen from the majority of other gases is its  
(a) Diamagnetism (b) Ferromagnetism  
(c) Paramagnetism (d) None of these.

### **Group- B**

2. (a) What do you mean by qualitative analysis? Justify the use of reference cell commonly used along with the measuring cell.  
[[CO5] (Remember/LOCQ), (Evaluate/HOCQ)]
- (b) Describe the working of Beckman's paramagnetic O<sub>2</sub> analyser with a neat sketch. Design the signal conditioning circuit to extract electrical output corresponding to the O<sub>2</sub> concentration. [[CO1] (Understand/LOCQ), (Create/HOCQ)]
- (c) Explain why O<sub>2</sub> lost its paramagnetic property at a higher temperature?  
[[CO1] (Analyse/IOCQ)]  
**(2 + 2) + (3 + 3) + 2 = 12**
3. (a) What do you mean by quantitative analysis. How do reference cell serves important role in analytical instruments?  
[[CO5] (Remember/LOCQ), (Evaluate/HOCQ)]
- (b) Describe the operation of a thermal conductivity analyser with neat sketch. Design the signal conditioning circuit necessary to extract analytical information from the above analyser. [[CO1] (Understand/LOCQ), (Create/HOCQ)]
- (c) How the heat of reaction method can be applied for analysis of combustible gas?  
[[CO5] (Analyse/IOCQ)]  
**(2 + 2) + (3 + 2) + 3 = 12**

### **Group - C**

4. (a) Demonstrate the operation of ISFET as a liquid analyser. How did the same ISFET can be used to detect multiple ions in an analytic solution?  
[[CO2] (Understand/LOCQ), (Create/HOCQ)]
- (b) Calculate the potential of the pH glass electrode when it is immersed in an analyte solution having pH value 5. Assume that the buffer solution inside the glass measurement bulb has a stable value of 7.0 pH and temperature of 25 degree Celsius. [[CO2] (Evaluate/HOCQ),]

- (c) Analyse the potential profiles across a glass membrane from the analyte solution to the internal reference solution. [(CO5) (Analyse/IOCQ)]  
**(4 + 2) + 3 + 3 = 12**
5. (a) Why measurement of liquid conductivity is important?  
[(CO2) (Remember/LOCQ)]
- (b) Design a measuring circuit for liquid conductivity system. Why is it better to use AC excitation rather than a DC excitation for the measuring circuit?  
[(CO5) (Create/HOCQ),(Analyse/IOCQ)]
- (c) How voltamogram can be used to extract qualitative and quantitative information about the analytic solution. [(CO2) (Apply/IOCQ)]  
**2 + (3 + 3) + 4 = 12**

### **Group - D**

6. (a) Sketch the optical path of a single beam instrument used in IR spectroscopy and precisely describe its function. [(CO3) (Understand/LOCQ)]
- (b) At 580 nm, which is the wavelength of its maximum absorption, the complex  $\text{Fe}(\text{SCN})^{2+}$  has a molar absorptivity of  $7.00 \times 10^3 \text{ L cm}^{-1} \text{ mol}^{-1}$ . Calculate the absorbance of a  $3.49 \times 10^{-5} \text{ M}$  solution of the complex at 580 nm in a 1.00-cm cell. Also compute transmittance of the solution. [(CO3) (Evaluate/HOCQ)]
- (c) How single-beam instrument differs from double-beam instruments for absorbance measurements. [(CO5) (Analyse/IOCQ)]  
**4 + 4 + 4 = 12**
7. (a) Describe operation of Time-of-Flight mass analysers with neat sketch.  
[(CO3) (Remember/LOCQ)]
- (b) Why atomic emission methods with an ICP source are better suited for multi-element analysis than flame atomic absorption methods?  
[(CO5) (Analyse/IOCQ)]
- (c) Aluminium is to be used as windows for a cell for X-ray absorption measurements with the Ag  $K\alpha$  line. The mass absorption coefficient for aluminium at this wavelength is  $2.74 \text{ cm}^2/\text{g}$ ; its density is  $2.70 \text{ g/cm}^3$ . What maximum thickness of aluminium foil could be used to fabricate the windows if no more than 2.8% of the radiation is to be absorbed by them?  
[(CO6) (Create/HOCQ)]  
**4 + 4 + 4 = 12**

### **Group - E**

8. (a) Define selectivity factor and retention time. Hence find the relation between them. [(CO4) (Remember/LOCQ), (Analyse/IOCQ)]
- (b) In HPLC, how Gradient Elution process differs from Isocratic Elution process?  
[(CO6) (Evaluate/HOCQ)]

(c) Describe operation of FID used in gas chromatography.

[(CO4) (Understand/LOCQ)]

**(2 + 3) + 4 + 3 = 12**

9. (a) With a schematic diagram, describe the operation of a differential refractive-index detector used in HPLC. [(CO4) (Understand/LOCQ)]

(b) How to interpret a chromatogram qualitatively as well as quantitatively.

[(CO5) (Analyse/IOCQ)]

(c) Consider a chromatogram of a mixture of species A and B with their retention time 13.3 minutes and 14.1 minutes respectively. Also the peaks of A and B have base widths 1.07 minutes and 1.16 minutes respectively. The unrestrained species takes 3.1 minutes to elute through the column. Compute the resolution and selectivity factor for the species. [(CO4) (Create/HOCQ)]

**5 + 3 + 4 = 12**

Cognition Level	LOCQ	IOCQ	HOCQ
Percentage distribution	35.42%	30.20%	34.38%

### Course Outcome (CO):

After the completion of the course students will be able to

1. Gain knowledge about gas analyzers.
2. Apply the liquid analysis techniques for analyzing liquids.
3. Acquire knowledge of UV, IR, X-ray and atomic mass spectroscopy.
4. Learn different chromatographic separation method used in industry and research purpose.
5. Select instrument for a particular analysis with some idea of its merits, demerits and limitations.
6. Learn operation of analytical tools that are used in hospitals for clinical analysis, drugs and pharmaceutical laboratories and above all for environmental pollution monitoring.

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

Department & Section	Submission Link
AEIE	<a href="https://classroom.google.com/c/NDA1MjA4MTI4MzE4/a/NDY0NTM1MTc1MDgw/details">https://classroom.google.com/c/NDA1MjA4MTI4MzE4/a/NDY0NTM1MTc1MDgw/details</a>