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 T and carrier gas flow rate F, the length of time that each component spends in the gas chromatograph column is called
(a) dead time (b) retarded time (c) delay time (d) retention time.

(ii) Which among the following elements has the highest thermal conductivity?
(a) Nitrogen (b) Oxygen (c) Hydrogen (d) Chlorine.

(iii) Typical detector of an IR gas analyser is
(a) Bolometer (b) Thermocouple (c) Thermopile (d) All of these.

(iv) Using the terms "oxidized" and "reduced" the potential of an electrode is given by $E = E_0 - \frac{0.0591}{n} \log \frac{[oxidized\ state]}{[reduced\ state]}$ originally derived by
(a) Nernst (b) Daimler (c) Einstein (d) Faraday.

(v) At 25°C, if a glass electrode with internal buffer solution of pH 7 when dip into an analyte solution of pH 6, the voltage generated across the glass membrane is
(a) +59.17 mV (b) -59.17 mV (c) +118.3 mV (d) -118.3 mV.

(vi) Dissolve CO_2 gas can be detected Molecular-Selective Electrode Systems with _____as sensing electrode.
(a) pCN (b) pH (c) pF (d) pS

(vii) Properties of Ion-Selective Membranes should has
(a) Minimal solubility (b) Electrical conductivity
(c) Selective reactivity with the analyte (d) All of these.

(viii) X-ray diffraction grating type monochromator obey the equation of
(a) Beer's (b) Bragg's (c) Michelson's (d) None of them.

(ix) The region of electromagnetic spectrum for nuclear magnetic resonance spectroscopy is
(a) Microwave (b) Infrared wave (c) Radio wave (d) UV rays.

(x) What will be the absorbance if %T = 80?
(a) 0.97 (b) -0.97 (c) 0.097 (d) -0.097.

Group - B

2. (a) What do you mean by qualitative and quantitative analysis? [(CO1)(Remember/LOCQ)]
(b) Using the necessary schematic diagram, describe the operation of a zirconia cell. How can the sample oxygen concentration be related to the cell emf? [(CO1)(Understand/LOCQ)]
(c) State the principle of working of the non-dispersive infrared gas analyser. [(CO1)(Analyse/IOCQ)]
4 + (4 + 2) + 2 = 12

3. (a) Why O_2 shows paramagnetic property? How this property can be applied for oxygen analysis? [(CO5)(Understand/LOCQ), (Apply/IOCQ)]
(b) Discuss how the Hays magnatherm analyser measures O_2 concentration. Why is it necessary to use a reference cell with this analyser? [(CO1)(Create/HOCQ), (Analyse/IOCQ)]
(c) Suggest a method of dissolve O_2 analyser. [(CO5)(Apply/IOCQ)]
(2 + 2) + (4 + 2) + 2 = 12

Group – C

4. (a) What do you mean by boundary potential of the glass electrode used for pH measurement? How will the polarity of the boundary potential change while immersing the glass electrode in an online stream whose pH is supposed to change from acidic to base? [(CO5)(Understand/LOCQ), (Evaluate/HOCQ)]
(b) Consider a glass electrode for measuring the pH of an analyte solution with a pH of 5. Assume the glass electrode's internal buffer solution has a pH of 7 and the working temperature is 25°C. Determine the emf generated across the glass membrane. [(CO2)(Evaluate/HOCQ),]
(c) What exactly do you mean by TDS in the context of liquid analysis? Give the names of two methods for measuring TDS. [(CO2)(Remember/LOCQ)]
(3 + 2) + 4 + 3= 12

5. (a) Draw a schematic diagram of a voltamometry instrumentation system and describe the same. [(CO2)(Understand/LOCQ)]
(b) Draw and analyse pulse polarogram. [(CO2)(Analyse/IOCQ)]
(c) Describe the phenomenon of concentration polarisation. [(CO2)(Remember/LOCQ)]
5 + 3 + 4 = 12

Group – D

6. (a) Describe various phenomena in which molecules of a species can absorb radiation. [(CO3)(Understand/LOCQ)]

(b) A solution containing the complex formed between Bi(III) and thiourea has a molar absorptivity of 9.32×10^3 L mol^{-1} cm^{-1} at 470 nm in a 1.00 cm cell. Concentration of the solution is 3.79×10^{-5} M. What is the percentage transmittance of the solution? [(CO3)(Evaluate/HOCQ)]

(c) State the advantages of FTIR instruments over dispersive spectrometers. [(CO5)(Analyse/IOCQ)]

6 + 4 + 2 = 12

7. (a) Explain operation of double beam flame atomic absorption spectrophotometers with suitable schematic diagram. [(CO3)(Understand/LOCQ)]

(b) What are some of the advantages of plasma sources over flame sources for emission spectrometry? [(CO5)(Analyse/IOCQ)]

(c) Aluminum is to be used as windows for a cell for X-ray absorption measurements with the Ag Kα line. The mass absorption coefficient for aluminum at this wavelength is 2.74 cm^2/g; its density is 2.70 g/cm^3. What maximum thickness of aluminum foil could be used to fabricate the windows if no more than 3.5% of the radiation is to be absorbed by them? [(CO6)(Evaluate/HOCQ)]

4 + 4 + 4 = 12

Group – E

8. (a) Describe the instrumental block diagram of a gas chromatography system. [(CO4)(Remember/LOCQ)]

(b) Where to use temperature programming during gas chromatographic run and how it works? [(CO6)(Analyse/IOCQ)]

(c) Describe the operation of Electron-Capture Detectors used in gas chromatography. [(CO4)(Understand/LOCQ)]

5 + 4 + 3 = 12

9. (a) Describe the principle of electrophoresis. [(CO4)(Understand/LOCQ)]

(b) Analyse how electrophoresis can be used to separate a mixture of DNA, RNA, and protein. [(CO5)(Analyse/IOCQ)]

(c) Consider a chromatogram of a mixture of species A, B and C with their retention time 10.0 minutes, 10.9 minutes and 13.4 minutes, respectively. Also the picks of A, B and C have base widths 0.76 minutes, 0.82 minutes and 1.06 minutes, respectively. The column length is 40 cm. Calculate the average number of plates and an average plate height for the column. [(CO4)(Evaluate/HOCQ)]

3 + 4 + 5 = 12

Cognition Level	LOCQ	IOCQ	HOCQ
Percentage distribution	48.96	23.96	27.08

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