ANALYTICAL INSTRUMENTATION
(AEIE 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 method is used in gas analysis to measure the thermal conductivity of a gas?
(a) Mass spectrometry (b) Thermal conductivity method
(c) Infrared spectroscopy (d) UV-Vis spectrometry

(ii) In a zirconia O_2 analyser, what is the role of stabilized zirconia?
(a) To prevent thermal conductivity
(b) To enhance electrical conductivity
(c) To maintain the stable cubic crystal structure
(d) To increase the optical transparency

(iii) Which material is widely used for homogeneous gas-permeable membranes?
(a) Polypropylene (b) Polytetrafluoroethylene
(c) Silicone rubber (d) PVC

(iv) The pH-sensitive glass electrode is made from which material?
(a) Polyethylene (b) Polystyrene
(c) Silver chloride (d) Thin glass membrane

(v) Which electrode is commonly used as a reference in pH measurement systems?
(a) Glass electrode (b) Calomel electrode
(c) ISFET electrode (d) Platinum electrode

(vi) In optical spectrometry, the sample is converted to gaseous atoms by
(a) X-ray radiation (b) Atomization
(c) Ionization (d) Mass-to-charge ratio analysis

(vii) The lamp used in atomic absorption spectroscopy as a radiation source is
(a) Tungsten filament lamp (b) Hollow-cathode lamp
(c) Halogen lamp (d) Sodium vapor lamp

(viii) The Beer's law in X-ray absorption is applied when
(a) X-rays are scattered
(b) The X-ray quantum is absorbed
(c) The electron is ejected from the outer shell
(d) Ionization does not occur

(ix) Which gas is most commonly used as the carrier gas in Gas Chromatography?
(a) Hydrogen (b) Nitrogen
(c) Argon (d) Helium

(x) What is the main purpose of the electron gun in a Scanning Electron Microscope?
(a) To detect secondary electrons (b) To create an image
(c) To produce a stream of electrons (d) To rotate the sample

Fill in the blanks with the correct word

(xi) In gas analysis, _____ is often used as a reference gas in paramagnetic oxygen analysers.

(xii) The energy of a photon is directly proportional to its _____ and inversely proportional to its wavelength.

(xiii) The two main types of columns used in gas chromatography are _____ columns and capillary columns.

(xiv) In Scanning Electron Microscopy (SEM), the _____ gun produces a stream of electrons necessary for imaging

(xv) The Nernst equation is used to relate the electrode potential to the _____ of the species in the solution.

Group - B

2. (a) Discuss analytical instrumentation and explain its importance in industrial applications. *[(CO1)(Analyse/IOCQ)]*
(b) What are the main characteristics of an ideal analytical instrument? Describe the basic components of an analytical instrument. *[(CO2)(Remember/LOCQ)]*
(c) Identify the challenges associated with measuring chemical compositions in gases? Explain how precision and accuracy differ in the context of analytical instrumentation. *[(CO1)(Apply/IOCQ)]*

2 + (2 + 3) + (3 + 2) = 12

3. (a) What is stabilized zirconia, and why is it important in the construction of zirconia O2 analysers? Discuss its role and benefits. *[(CO3)(Remember/LOCQ)]*
(b) Explain the principle of operation of a thermal conductivity analyser and describe how it is used to determine the composition of gas mixtures. *[(CO4)(Evaluate/HOCQ)]*
(c) Interpret the advantages and limitations of using thermistors as heat-sensing elements in thermal conductivity gas analysers. *[(CO2)(Understand/LOCQ)]*
(d) Identify the principle of the paramagnetic method for oxygen analysis and explain how Beckman's Paramagnetic O2 Analyser utilizes this principle. *[(CO2)(Apply/IOCQ)]*

(2 + 2) + (2 + 2) + 2 + 2 = 12

Group - C

4. (a) What is the function of a reference electrode in electrochemical cells? *[(CO3)(Remember/LOCQ)]*

   (b) Discuss the construction of a silver-silver chloride electrode. How does a gas-sensing probe measure dissolved gases? *[(CO4)(Create/HOCQ)]*

   (c) Explain how the potential of an electrochemical cell is related to the free energy of the reaction. *[(CO2)(Understand/LOCQ)]*

   (d) What is the Nernst equation and how is it used in electroanalytical chemistry? *[(CO2)(Remember/LOCQ)]*

2 + (3 + 3) + 2 + 2 = 12

5. (a) Explain the significance of Faraday's constant in electrochemistry. *[(CO3)(Analyse/HOCQ)]*

   (b) What role does potassium nitrate play as a substitute in salt bridges? *[(CO4)(Remember/LOCQ)]*

   (c) What are the primary applications of voltammetry in liquid analysis? Discuss the applications of ISFETs in modern analytical instrumentation. *[(CO2)(Apply/IOCQ)]*

   (d) How does the potential of a cell change with the activity of reactants? *[(CO2)(Apply/IOCQ)]*

3 + 3 + (2 + 2) + 2 = 12

Group - D

6. (a) Define atomization and explain its significance in atomic spectrometry. *[(CO3)(Analyse/HOCQ)]*

   (b) Compare and contrast atomic absorption and emission spectra. *[(CO4)(Remember/LOCQ)]*

   (c) Describe the process of fluorescence in atomic spectrometry. *[(CO2)(Apply/IOCQ)]*

   (d) Explain the role of a nebulizer in atomization methods. *[(CO2)(Apply/IOCQ)]*

3 + 3 + 3 + 3 = 12

7. (a) Explain the working principle of a mass spectrometer with a suitable block diagram. *[(CO3)(Analyse/HOCQ)]*

   (b) List the major applications of mass spectrometer in industry and research. *[(CO4)(Remember/LOCQ)]*

   (c) Describe the three electrode geometry of Paul Trap. *[(CO2)(Apply/IOCQ)]*

   (d) Which part of the mass spectrometer is working as a mass analysing unit? Discuss the issues associated with the miniaturisation of ion traps. *[(CO2)(Apply/IOCQ)]*

2 + 2 + 2 + (2 + 4) = 12

Group - E

8. (a) What is the role of the stationary phase in gas chromatography? *[(CO3)(Analyse/HOCQ)]*

   (b) Explain the concept of retention time in gas chromatography. *[(CO4)(Remember/LOCQ)]*

   (c) How does the Flame Ionization Detector (FID) work in gas chromatography. *[(CO2)(Apply/IOCQ)]*

(d) What is the importance of the carrier gas in gas chromatography, and how is its flow rate controlled. *[(CO2)(Apply/IOCQ)]*

2 + 2 + 2 + (3 + 3) = 12

9. (a) Explain the role of the mobile and stationary phases in HPLC. Describe the function of the pump in an HPLC system. *[(CO3)(Analyse/HOCQ)]*

(b) What are the key differences between isocratic and gradient elution in HPLC? *[(CO4)(Remember/LOCQ)]*

(c) What are the advantages of using a UV-visible absorption detector in HPLC? *[(CO2)(Apply/IOCQ)]*

(d) What are the typical applications of HPLC in pharmaceuticals and environmental analysis? *[(CO2)(Apply/IOCQ)]*

(3 + 3) + 2 + 2 + 2 = 12

Cognition Level	LOCQ	IOCQ	HOCQ
Percentage distribution	30.20	42.80	27.08