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) In a thermal conductivity analyser, which materials are commonly used for constructing filaments?
(a) Gold and Silver (b) Platinum and Tungsten
(c) Copper and Aluminium (d) Iron and Nickel.

(ii) What is the principle behind the paramagnetic method of oxygen analysis?
(a) Chemical reactivity (b) Electrical conductivity
(c) Magnetic susceptibility (d) Optical absorption.

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

(iv) What is the role of potassium chloride in a calomel electrode?
(a) Acts as a reactant
(b) Maintains the concentration of chloride ions
(c) Acts as an internal reference
(d) Reacts with silver ions.

(v) In atomic fluorescence spectroscopy, radiation emitted is measured at an angle of
(a) 45° (b) 60° (c) 90° (d) 180°

(vi) 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.

(vii) Which detector is used in atomic X-ray spectrometry to measure radiation?
(a) Photodiode (b) Photomultiplier tube
(c) Proportional counter (d) CCD array.

(viii) What is the primary role of the mobile phase in Gas Chromatography?
(a) To act as a stationary phase
(b) To separate the analytes
(c) To carry the sample through the column
(d) To detect the analytes.

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

(x) Which principle is used in electrophoresis for the separation of molecules?
(a) Molecular weight (b) Charge-to-size ratio
(c) Absorbance (d) Refractive index.

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 standard reduction potential for the Ag/AgCl electrode at 25°C is _____ volts.

(xiii) 3D Ion traps are known as _____ Trap.

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

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

Group - B

2. (a) Explain the working principle of a Wheatstone bridge in a thermal conductivity analyser. *[(CO3)(Understand/LOCQ)]*
(b) How do changes in gas concentration affect the filaments in a thermal conductivity analyser? *[(CO4)(Remember/LOCQ)]*
(c) Contrast how the thermal conductivity method can be used to measure hydrogen in blast furnace gases. *[(CO2)(Apply/IOCQ)]*
(d) Why are platinum and tungsten commonly used in the filaments of thermal conductivity analysers? *[(CO2)(Remember/LOCQ)]*

3 + 3 + 4 + 2 = 12

3. (a) What makes oxygen paramagnetic and how does this property help in oxygen analysis? What are the limitations of the paramagnetic method for oxygen analysis? *[(CO3)(Remember/LOCQ)]*
(b) Explain how paramagnetic analysers compensate for environmental variations. *[(CO4)(Understand/LOCQ)]*
(c) Discuss the significance of measuring oxygen levels in industrial processes using the paramagnetic method. *[(CO2)(Create/HOCQ)]*
(d) How does the null-type method work in a paramagnetic oxygen analyser? *[(CO2)(Remember/IOCQ)]*

(3 + 2) + 3 + 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) Discuss the limitations of measuring absolute electrode potentials. *[(CO3)(Analyse/HOCQ)]*

   (b) What is the function of a salt bridge in reducing liquid-junction potential? *[(CO4)(Remember/LOCQ)]*

   (c) Assess how membrane electrodes allow selective ion detection. What are the key properties of ion-selective membranes? *[(CO2)(Evaluate/HOCQ)]*

   (d) Explain the working principle of a calomel electrode. *[(CO2)(Understand/LOCQ)]*

2 + 2 + (2 + 3) + 3 = 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) Describe the working principle of a UV/ visible spectroscopy. *[(CO3)(Analyse/HOCQ)]*

   (b) Define the term “Transmittance” in this type of spectroscopy. *[(CO4)(Remember/LOCQ)]*

   (c) Distinguish between dual channel spectrophotometer from dual beam. *[(CO2)(Apply/IOCQ)]*

   (d) How is the correct wavelength selected in spectrophotometry? *[(CO2)(Apply/IOCQ)]*

3 + 2 + 4 + 3 = 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) What is the function of the electron gun in SEM? *[(CO3)(Analyse/HOCQ)]*

(b) Explain the role of the lenses in focusing the electron beam in SEM. *[(CO4)(Remember/LOCQ)]*

(c) What are Everhart-Thornley detectors, and how do they contribute to imaging in SEM? *[(CO2)(Apply/IOCQ)]*

(d) Explain the difference between secondary electrons and backscattered electrons in SEM. *[(CO2)(Apply/IOCQ)]*

4 + 3 + 3 + 2 = 12

Cognition Level	LOCQ	IOCQ	HOCQ
Percentage distribution	38.54	33.33	28.13