

**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 chromatogram, the peak base width is expressed in
(a) Centimetre (b) Meter
(c) Minute (d) Micrometer.
- (ii) The susceptibility of oxygen is
(a) Less than 1 (b) Equal to 1
(c) Greater than 1 (d) Equal to 0.
- (iii) The pH value of a liquid indicates
(a) Hydroxyl ion concentration (b) Hydrogen ion concentration
(c) Oxygen ion concentration (d) None of (a), (b) & (c).
- (iv) The diamagnetic gas used in thermomagnetic type oxygen analyzer is
(a) Hydrogen (b) Nitrogen
(c) Oxygen (d) Carbon dioxide.
- (v) Gel Permeation chromatography is the classification of
(a) Gas solid chromatography (b) Gas liquid chromatography
(c) High pressure liquid chromatography (d) Liquid chromatography.
- (vi) If I_0 & I indicate the intensities of light incident and emitted by the sample respectively, then the Transmittance T is expressed as
(a) $T = I_0 / I$ (b) $T = I / I_0$
(c) $T = \log_{10}(I_0 / I)$ (d) $T = \log_{10}(I / I_0)$.
- (vii) In molecular absorption spectroscopy absorption (A) of radiation depends on the path length (b) of radiation in the medium and concentration(c) of the medium by the following manner where 'a' is the absorptivity
(a) $A = ab/c$ (b) $A = abc$
(c) $A = (a+b)/c$ (d) $A = bc/a$.

- (viii) If the wavelength of electromagnetic radiation increases, the energy of it
 (a) Decreases (b) Remains constant
 (c) Increases (d) Becomes infinite.
- (ix) The detector used in FTIR spectrometry is
 (a) Goniometer (b) Scintillation counter
 (c) Photomultiplier tube (d) Pyroelectric detector.
- (x) The Zirconia cell is used to determine the
 (a) Density of a fluid
 (b) Moisture content of a gas
 (c) Oxydation – reduction potential of an electrolyte
 (d) Quantitative analysis of oxygen.

Fill in the blanks with the correct word

- (xi) Relative value of susceptibility of N₂ considering O₂ as 100 is _____.
- (xii) In gas chromatography the expression of column resolution is _____.
- (xiii) The wavelength range of ultra violet radiation is _____.
- (xiv) The unit of thermal conductivity of a gas is _____.
- (xv) The mass analyser used in ion trap mass spectrometer is _____.

Group - B

2. (a) What is thermal conductivity of a gas mixture? How it is related to the concentration of the individual gas components? [[CO1](Analyse/IOCQ)]
- (b) With a neat diagram explain the operation of thermal conductivity analyzer for measuring a gas component in a binary gas mixture. How the calibration curve is prepared for the instrument response? [[CO1](Analyse/IOCQ)]
- (c) What is resistance to thermal conductivity sensitivity of thermal conductivity analyzer? [[CO1](Evaluate/HOCQ)]
- (2 + 2) + (5 + 1) + 2 = 12**
3. (a) What is the principle of paramagnetic type instrument for the analysis of oxygen? [[CO1](Understand/LOCQ)]
- (b) Draw the set up for deflection type paramagnetic oxygen analyzer and describe the working of it. [[CO1](Analyse/IOCQ)]
- (c) What are the precautions taken for the deflection type paramagnetic instrument while making quantitative analysis of oxygen? [[CO1](Remember/LOCQ)]
- 2 + 6 + 4 = 12**

Group - C

4. (a) Show and explain the instrumental set up of polarographic method with dropping mercury electrode for liquid analysis. [[CO2](Analyse/IOCQ)]

- (b) How quantitative analysis of the components can be made from the polarogram obtained for the solution having copper, cadmium and zinc? [[CO2) (Apply/HOCQ]]
- (c) What are the applications of polarography? [[CO2)(Remember/LOCQ]]
- 6 + 3 + 3 = 12**
5. (a) What is the principle of pH measurement? [[CO6)(Analyse/IOCQ]]
- (b) Why does asymmetry potential arise in glass electrode and how it is compensated? [[CO6)(Analyse/IOCQ]]
- (c) Draw and describe the features of the electrodes used in commercially available pH measuring arrangement. [[CO6)(Understand/LOCQ]]
- 3 + 3 + 6 = 12**

Group - D

6. (a) With a neat diagram explain the operation of a dual beam type spectrophotometer for the analysis of liquid sample concentration. [[CO3)(Analyse/IOCQ]]
- (b) How ionization process is done by using ion trap in mass spectrometry? Name at least two types of ion traps. [[CO3)(Apply/HOCQ]]
- (c) What are x-ray emission and x-ray fluorescence spectrography? [[CO3)(Understand/LOCQ]]
- 4 + (4+2) + 2 = 12**
7. (a) Specify the application areas of IR spectroscopy. [[CO3)(Remember/LOCQ]]
- (b) State the criteria/ requirements for a compound to absorb IR Radiation. [[CO3)(Analyse/IOCQ]]
- (c) How does a prism monochromator work as a wavelength selector unit in molecular absorption spectroscopy? [[CO3)(Understand/LOCQ]]
- 4 + 5 + 3 = 12**

Group - E

8. (a) What are distribution coefficient and capacity factor in chromatography? [[CO4)(Understand/LOCQ]]
- (b) Explain briefly gas-liquid and gas-solid chromatographic methods. [[CO4) (Understand/LOCQ]]
- (c) A gas chromatogram of a mixture containing benzene, anthracene and air (not retarded on column) was obtained. The retention time for the above components are 3.24 min, 5.73 min and 0.25 min respectively. What is the capacity factor for benzene? Also find the relative retention of anthracene with respect to benzene. [[CO4)(Evaluate/HOCQ]]
- 4 + 4 + 4 = 12**
9. (a) Draw the block diagram of high pressure liquid chromatography. Mention different components of it. Explain how sample injection system works for this chromatographic technique. [[CO4)(Analyse/IOCQ]]

(b) What are gel permeation and ion-exchange type liquid chromatography?

[[CO4](Remember/LOCQ)]

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

Cognition Level	LOCQ	IOCQ	HOCQ
Percentage distribution	37	39	24

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.*