Answer FIVE questions; answer Section A, TWO questions from Section B, and ONE question from Section C, and attempt a fifth question from either Section B or C.

Use separate answer books for each Section.
All questions carry equal marks.

Section A

Attempt any TEN parts. All parts carry equal marks.

Q1. (i) Identify the method of analysis described by the following:
A method which involves exciting a sample at one wavelength, inducing absorption of electromagnetic radiation and observing and measuring emission at 90°.

(ii) MgCl₂ has a concentration of 0.054 M in the ocean. How many grams of MgCl₂ are present in 25 ml of sea water? Mg = 24.3 a.m.u. Cl = 35.5 a.m.u.

(iii) If a constituent in a sample is classified as minor, what percentage of it is present?

(iv) Distinguish between the terms overtone and combination with respect to infrared spectroscopy.

(v) An infrared spectrum may often have fewer bonds that the expected number of normal modes of vibration. Give two reasons to account for this.

(vi) Name two atomic spectroscopic methods, one based on emission and one on absorption.

(vii) What is an ion selective electrode?

(viii) Explain the letters ‘GF-AAS’

(ix) Draw a simple energy level diagram, which illustrates the differences in energy between rotational, vibrational and electronic energy levels.

(x) What is the absorbance of a solution, which transmits 65% of the incident radiation?

(xi) Give the circuit diagram symbol for an n.p.n. transistor and draw a diagram illustrating its biasing arrangements.
(xii) State two properties of an ideal detector.

(xiii) A reflection grating has 1000 lines per millimetre. Calculate the second order resolving power if 8 cm of the grating is illuminated.

(xiv) Give two reasons why stray (spurious) wavelengths occur in the output beam of a monochromator.

Section B

Q2. (a) Absorption of ultraviolet (UV) radiation by a molecule occurs if it contains a chromophore. If the molecule has more than one chromophore which are conjugated together it may result in a bathochromic shift and hyperchromism.

(i) Explain the underlined terms.

(ii) How can the effects of conjugation as outlined above be explained? (9 marks)

(b) The UV spectrum of benzonitrile shows a primary absorption bond at 224 nm and a secondary bond at 271 nm. If a solution of benzonitrile in water with a concentration of $1 \times 10^{-4}$ mol dm$^{-3}$, is examined at 224 nm and the absorbance determined to be 1.30, what is the molar absorptivity of this absorption band if the measurements are made in a 1 cm cell? (4 marks)

(c) Deviations from Beer’s Law occur at both high and low analyte concentrations. Identify two and explain one such deviation in each scenario. (7 marks)

Q3. (a) Compare and contrast gas chromatography (GC) and high performance liquid chromatography (HPLC) under the following headings:

(i) sample size and destruction

(ii) quantitative applications

(iii) columns

(iv) ability to deal with non volatile and thermally unstable samples. (8 marks)

(b) Describe the fundamental differences between adsorption and partition chromatography. Diagrams required. (6 marks)
(c) For a chromatogram containing three peaks, the relative areas and relative detector responses are given in the following table:

<table>
<thead>
<tr>
<th>Component no:</th>
<th>Relative Peak Areas</th>
<th>Relative detected response</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>16.4</td>
<td>0.60</td>
</tr>
<tr>
<td>2</td>
<td>45.2</td>
<td>0.78</td>
</tr>
<tr>
<td>3</td>
<td>30.2</td>
<td>0.88</td>
</tr>
</tbody>
</table>

Calculate the percentage of each component. (6 marks)

Q4. Attempt three of the following:

(i) Define the following terms used in HPLC.
   (a) gradient elution
   (b) isocratic elution
   (c) reversed–phase packing
   (d) normal-phase packing
   (e) bonded-phase packing

(ii) Describe the physical differences between open tubular and packed columns. What are the advantages and disadvantages of each?

(iii) Describe how an infrared spectrum of solid, liquid and gaseous samples may be obtained. Include in your answer comments on:
   (a) sample size
   (b) cell materials and cell size
   (c) suitable solvents and reagents

(iv) Chemical and physical interferences are two of the interferences associated with flame atomisation methods in atomic spectroscopy. Discuss both in detail. Name two other interferences.

(v) Glass membrane electrodes are one of four types of ion selective electrode. Discuss briefly. Name the three other types. (20 marks)
Q5. (a) Identify three characteristics of the mobile phase used in gas chromatography. Give an example. (4 marks)

(b) The data in the following table were obtained during a G.C. determination of a C₁₀ hydrocarbon, with a closely related compound added to each standard and to the unknown as an internal standard. The unknown was prepared by taking 12.5 cm³ of the original sample solution and diluting it to 50 cm³ in a volumetric flask. This was done in triplicate.

<table>
<thead>
<tr>
<th>% Analyte</th>
<th>Analyte Peak Height/mm</th>
<th>Internal Std. Peak Height/mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>18.8</td>
<td>50.0</td>
</tr>
<tr>
<td>0.10</td>
<td>48.1</td>
<td>64.1</td>
</tr>
<tr>
<td>0.15</td>
<td>63.4</td>
<td>55.1</td>
</tr>
<tr>
<td>0.20</td>
<td>63.2</td>
<td>42.7</td>
</tr>
<tr>
<td>0.25</td>
<td>93.6</td>
<td>53.8</td>
</tr>
<tr>
<td>Unknown (1)</td>
<td>58.9</td>
<td>49.4</td>
</tr>
<tr>
<td>(2)</td>
<td>57.9</td>
<td>49.4</td>
</tr>
<tr>
<td>(3)</td>
<td>65.8</td>
<td>60.4</td>
</tr>
</tbody>
</table>

(i) Construct an appropriate calibration curve.
(ii) Hence, determine the concentration of the unknown in the original sample solution.
(iii) Explain the function of the internal standard. Comment on the data obtained for the unknown analysis. (12 marks)

(c) The standards listed in the table were prepared by dilution of a 0.8% C₁₀ stock solution. What volume of this stock is required to prepare:
(i) 50 cm³ of the 0.10% standard solution?
(ii) 25 cm³ of the 0.25% standard solution? (4 marks)
Section C

Q6. (a) Photosensitive devices respond to light while photo emissive devices radiate light. Give one example of each of the above types of optoelectronic device. (2 marks)
Write brief notes on each device you have named, stating:
(i) the circuit diagram symbol
(ii) how the device works
(iii) an application or use of the device. (6 marks)

(b) Draw a block diagram of a stabilised d.c. power supply unit. (2 marks)

Draw a detailed circuit diagram of the rectifier stage and explain how it works. (7 marks)

Sketch the input and output voltage waveforms. Comment on the output voltage waveform. (3 marks)

Q7. In a commercial uv-visible spectrophotometer, wavelength selection is achieved by means of a monochromator.

(a) State two advantages of a monochromator as compared with a filter. (2 marks)

(b) Draw a diagram of the optical arrangement of a monochromator you have studied. List the function of each component. (8 marks)

(c) Light is incident on a reflection grating at an angle of 30º and is reflected on the opposite side of the grating normal at an angle of 20º.
If the grating has 1500 grooves per mm, calculate the first order wavelength selected. (6 marks)

(d) Write a brief note on the manufacture of reflection gratings. (4 marks)