

Cork Institute of Technology

Higher Certificate in Science in Applied Biology – Award
(National Certificate in Science in Applied Biology – Award)
(NFQ – Level 6)

Summer 2005

Instrumental Analysis

(Time: 3 Hours)

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.

Examiners: Dr. R. Hourihane
Ms. C. Devaney
Dr. T. Beresford

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) MgCl_2 has a concentration of 0.054 M in the ocean. How many grams of MgCl_2 are present in 25 ml of sea water? $\text{Mg} = 24.3 \text{ a.m.u.}$ $\text{Cl} = 35.5 \text{ a.m.u.}$
- (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 bands than 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 bathchromic 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 band at 224 nm and a secondary band at 271 nm. If a solution of benzonitrile in water with a concentration of $1 \times 10^{-4} \text{ mol dm}^{-3}$, 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 distruction
 - (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:

<i>Component no:</i>	<i>Relative Peak Areas</i>	<i>Relative detected response</i>
1	16.4	0.60
2	45.2	0.78
3	30.2	0.88

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.

% Analyte	Analyte Peak Height/mm	Internal Std. Peak height/mm
0.05	18.8	50.0
0.10	48.1	64.1
0.15	63.4	55.1
0.20	63.2	42.7
0.25	93.6	53.8
Unknown (1)	58.9	49.4
(2)	57.9	49.4
(3)	65.8	60.4

- (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 30° and is reflected on the opposite side of the grating normal at an angle of 20° .
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)