

Cork Institute of Technology

Higher Certificate in Science in Applied Biology – Award
(National Certificate in Science in Applied Biology – Award)

(NFQ – Level 6)

Autumn 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: Ms. C. Devaney
Dr. R. Hourihane
Dr. Tom Beresford

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

Section A - compulsory

Attempt any Ten parts.

All parts carry equal marks.

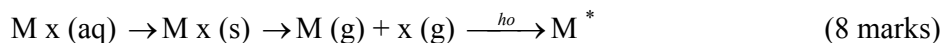
- Q1. (i) Sampling is the process of selecting a representative bulk sample from the lot.
Explain the underlined terms.
- (ii) A sample weighs 1.4352g and contains 0.0101g of potassium. What is the content of potassium in parts per million?
- (iii) State whether the following solutions are coloured or colourless.
- (v) A solution with λ max 555nm.
- (vi) A solution with λ max 250nm.
- (iv) Explain the term chromophore and give an example.
- (v) Indicate the order in which the following compounds would be eluted from the HPLC column containing reversed – phase packing.

benzene, diethyl ether, hexane

- (vi) Name two common detectors used in HPLC.
- (vii) Illustrate a simple diagram of the technique of adsorption chromatography.
- (viii) List two properties of the carrier gas used in gas chromatography.
- (ix) How many g/dm^3 of Na Cl are contained in a 0.250 mol/dm^3 solution.
- Na = 23.00 Cl = 35.5
- (x) When you double the wavenumber of electromagnetic radiation you the energy.
- (xi) List two requirements in the design of a laser.
- (xii) Name the sources used to provide (i) visible and (ii) infrared radiation in spectrophotometers. Give approximate wavelength ranges for the sources you named.
- (xiii) Draw circuit diagrams showing a pn junction diode under (i) forward and (ii) reverse bias conditions.
- (xiv) Calculate the wavelength selected in first order by an interference filter with the following specifications: thickness of dielectric layer, (magnesium fluoride) = 1694 nm. Refractive index of magnesium fluoride = 1.34.

Section B

- Q2. (a) The following equation summarises the process of atomic absorption spectroscopy, (AAS). Describe each of the steps in words, identifying each of the symbols.



- (b) Background correction is essential in most atomic spectroscopic methods.
- (i) What is meant by background correction.
- (ii) Describe two methods by which it may be achieved. (8 marks)
- (c) Name and describe briefly a common (simple) emission spectroscopic technique with a similar atomisation temperature, to (AAS). (4 marks)

- Q3. (a) Describe the process of gas chromatography (GC) from sample introduction to elution.
Supplement your answer with a schematic diagram of the instrument. (10 marks)

- (b) Outline how solid, liquid and gaseous samples may be introduced into a gas chromatography. (5 marks)
- (c)
- (i) How many theoretical plates produce a chromatography peak eluting at 12.83 mins with a width at half height of 8.7s?

Hence or otherwise calculate the efficiency (E) of the column.

- (ii) If the length of the column is 15.8cm, calculate the height equivalent to a theoretical plate. (HETP) (5 marks)

- Q4. Attempt three of the following:

- (i) A solution containing $3.47 \times 10^{-3}M$ (analyte x) and $1.72 \times 10^{-3}M$ (internal std. S) gave peak areas of 3473 and 10,222 respectively, in a chromatographic analysis. Then 1.00ml of $8.47 \times 10^{-3}M$ (S) was added to 5.00ml of unknown X, and the mixed solution was diluted to 10.0ml. This solution gave peak areas of 5428 and 4431 for X and S, respectively.

Find the concentration of X in the original unknown.

- (ii) Describe the method of thin layer chromatography. (TLC). In your answer explain the reference points on a typical TLC plate; i.e.

- Sample application point
- Solvent front
- Retardation factor

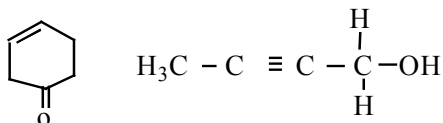
A diagram is required.

- (iii) Describe in detail the reasons why broadening of an initially sharp band of solute occurs as it moves through a chromatography column.

Diagrams to illustrate the various broadenings required.

- (iv) (a) For the two compounds illustrated below, identify and name the functional groups which may give rise to a signal in an infrared spectrum.

- (b) Give the approximate wavenumber position for these signals. Hence or otherwise, name the sections of the mid-infrared region where these signals occur.



- (v) Give an expression for Beer's Law, explain each of the symbols; giving the unit where appropriate. A 7.50×10^{-5} mol/L solution of potassium permanganate has a transmittance of 36.4 % when measured in a 1.05 cm cell at a wavelength of 525nm. Calculate

- (a) the absorbance of this solution
 (b) the molar absorptivity of KMnO_4

Q5. (a) Determination of a metal by ultraviolet visible spectrometry involves the formation of a metal chelate or complex. Identify and discuss briefly three important characteristics an ideal reagent must possess. (7 marks)

(b) A nitrite (NO_2^-) analysis of aquarium water was conducted according to the following procedure. For each analysis, 10.00ml of standard or unknown was diluted up to 100.00ml with water. Then to 50.00ml of each diluted solution 2.00ml of colour forming reagent was added. After 10 mins, the absorbance at 543nm, of each solution was determined with a 1.000cm path length cuvette. The unknown was analysed in triplicate. The results are summarised in the following table.

(i) Fill in the corrected absorbance, which is the measure of absorbance minus the average blank absorbance.

Copy the table into your answer book when completed.

(ii) Construct an appropriate calibration plot and find the
 (1) ppm of nitrate nitrogen and
 (2) molar concentration of nitrite in the aquarium

Sample	Absorbance @ 543nm	Corrected Absorbance
Blank	0.022	-
Blank	0.024	-
Standards		
0.538 ppm	0.121	
1.076 ppm	0.219	
2.152 ppm	0.413	
3.228 ppm	0.600	
4.034 ppm	0.755	
unknown	0.333	
unknown	0.339	
unknown	0.338	

(13 marks)

Section C

- Q6. (a) (i) Draw a diagram illustrating the optical arrangement of an interferometer. (4 marks)
- (ii) State two advantages of fourier transform IR spectrophotometers. (2 marks)
- (b) (i) List five properties of an ideal detector. (5 marks)
- (ii) Name the detector used in a commercial UV – visible spectrophotometer. (2 marks)
- (ii) Discuss the performance of this detector with regard to “ideal” behaviour (refer in your answer to (b) (i) above). (5 marks)
- Q7. (a) (i) Distinguish between intrinsic and extrinsic semiconductor material. (2 marks)
- (ii) Give a brief account of the mechanism of conduction in n type semiconductor material. (6 marks)
- (b) (i) Draw a circuit diagram of a common emitter voltage amplifier circuit. (5 marks)
- (ii) State the function of each component in the circuit. (5 marks)
- (iii) Explain the term, “voltage gain”. (2 marks)