

Cork Institute of Technology  
Higher Certificate in Science in Applied Biology – Award

(NFO Level 6) – Old Syllabus

Summer 2006

**Instrumental Analysis**

(Time: 3 Hours)

Instructions:

Answer FIVE questions. Answer Section A.  
Answer TWO questions from Section B.  
Answer ONE question from Section C. Answer  
ONE other question from either Section B or C.

Examiners: Dr. R. Hourihane  
Ms. C. Devaney  
Prof. R. Fitzgerald

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

**Section A**

Q1. Attempt any TEN parts. All parts carry equal marks.

- (i) State whether the following solutions are coloured or colourless.
  - (a) solution with  $\lambda_{\max}$  550nm
  - (b) solution with  $\lambda_{\max}$  250nm.
- (ii) What are the normal  $x$  and  $y$  ordinates used in
  - (a) UV spectrum?
  - (b) IR spectrum?
- (iii) Describe two basic differences between atomic emission and atomic absorption spectroscopy.
- (iv) Describe the difference between gel filtration and gel permeation chromatography.
- (v) The mobile phase in HPLC can consist of a mixture of solvents. Illustrate a three solvent system.
- (vi) A chromatogram of a mixture of species A, B, C and D provides the following data:

Component	time/min	Width of Peak Base/mm
A	5.4	0.41
B	13.3	1.07
C	14.1	1.16
D	21.6	1.72

Calculate the number of plates/efficiency from the data.

- Q1.(vii) If drinking water contains 1.5 ppm of Na F. How many litres of water can be fluoridated with 454g (1lb) of Na F?
- (viii) Calculate the molarity of a sample that contains 6.00 g NaCl in 200mL of solution.
- (ix) List, by name and wavenumber range, the four divisions into which the mid infrared region can be divided.
- (x) List three limitations associated with HPLC.
- (xi) Draw a block diagram of a stabilised dc power supply unit.
- (xii) Draw a diagram showing the biasing arrangement of an npn transistor.
- (xiii) Give two reasons why stray radioations occur in the out-put beam of a monochromator.
- (xiv) A reflection grating has 1000 lines per millimeter. Calculate the first order resolving power if 3 cm of the grating is illuminated. (20 marks)

## Section B

- Q2. (a) High performance liquid chromatography (HPLC) and traditional liquid chromatography both fall under the general heading of liquid chromatography, but they differ significantly.  
Identify four differences between them. (4 marks)
- (b) Bonded phases in HPLC offer a wide range of stationary phases and polarities.
- (i) What is meant by the term Bonded Stationary Phase? (1 mark)
- (ii) Describe, in detail, how bonded stationary phases are made illustrating the range of polarities available. (6 marks)
- (iii) List three advantages offered by bonded stationary phases over liquid solid chromatography (LSC). (3 marks)
- (c) Uv/visible absorbance detectors are by far the most popular detectors in HPLC. List three categories of this detector. Describe their mode of operation. (6 marks)

- Q3. (a) (i) State the difference between transmittance, absorbance and molar absorptivity.  
 (ii) Which one is proportional to concentration?  
 (iii) Name and write the law which summaries the relationship referred to in (ii).  
 (6 marks)
- (b) Apotransferrin has a molar absorptivity of  $8.83 \times 10^4 \text{ M}^{-1} \text{ cm}^{-1}$  at 280 nm.  
 (i) Find the concentration of apotransferrin in water if the absorbance is 0.244 in 0.100cm cell.  
 (ii) If the molecular weight of apotransferrin is 81,000g/mol express the concentration calculated in (i) above in g/L. Hence, or otherwise, calculate the absorptivity.  
 (6 marks)
- (c) Complexation reagents/metal chelating agents are used in the spectroscopic determination of transition metals.  
 (i) Explain the function of such reagents. (2 marks)  
 (ii) Identify and explain in detail four properties of an ideal reagent. (4 marks)  
 (iii) Give two examples of these reagents and the metal they are most likely to complex.  
 (2 marks)

Q4. Attempt three of the following:

- (a) Write a detailed note on the interpretation of infrared spectra.
- (b) 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.
- (c) Sampling and sample preparation are two important related steps in the analytical process. Describe both in detail.  
 Diagrams and examples are required.
- (d) Write a note explaining the term Thin Layer Chromatography (TLC).  
 In your discussion outline:  
 (i) Whether the method is qualitative or quantitative?  
 (ii) How components are measured?  
 (iii) The mechanism by which the technique operates.
- (e) Describe two methods by which a sample may be introduced onto a column in gas chromatography. Include in your discussion any limitations associated with each method and how they may be minimised/eliminated. (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 C<sub>7</sub> 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 cm<sup>3</sup> of the original sample solution and diluting it to 50 cm<sup>3</sup> 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% C<sub>7</sub> stock solution.  
What volume of this stock is required to prepare

- (i) 50 cm<sup>3</sup> of the 0.10% standard solution?
- (ii) 25 cm<sup>3</sup> of the 0.25% standard solution? (4 marks)

## Section C

- Q6. (a) (i) What is the purpose of a rectifier circuit? (2 marks)
- (ii) Explain, with the aid of a diagram, the operation of a Bridge Rectifier Circuit.  
(Give input and output voltage waveforms). (8 marks)
- (iii) Comment on the quality of the output of the above circuit and state two ways in which it could be improved. (4 marks)
- (b) Write a brief note on a photodiode. Comment on the suitability of this detector for the detection of u.v. – visible radiation. (6 marks)

- Q7. (a) Wavelength selection may be achieved using the following:
- (i) an interference filter (2 marks)
- (ii) a monochromator (3 marks)
- (iii) an interferometer (3 marks)

Draw labelled diagrams of each of the above.

Discuss briefly the relative merits of each in the selection of uv – visible and I.R. radiation in spectrophotometers. (6 marks)

- (b) A reflection grating has 1000 lines or grooves per millimeter. What first and second order wavelengths will appear for an angle of incidence,  $i = 40^\circ$  and an angle of reflection,  $r = 5^\circ$ ? (Both angles are on the same side of the grating normal) (6 marks)