Cork Institute of Technology

Higher Certificate in Science in Applied Biology (Old Syllabus)– Award

(NFQ Level 6)

Autumn 2006

Instrumental Analysis

(Time: 3 Hours)

Instructions
Answer FIVE questions.
Answer Section A. Attempt TWO questions from Section B, and ONE question from Section C.
Attempt a FIFTH question from either Section B or C.
Use separate answer books for each Section.
All questions carry equal marks.

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

Section A

Q1. Attempt any ten parts. All carry equal parts.

(i) Sampling is the process of selecting a representative bulk sample from the lot. Explain the underlined terms.

(ii) What is meant if something is said to be a trace constituent in a sample?

(iii) How many g/dm^3 of NaCl are contained in a 0.250 mol/dm^3 solution?

Na = 23.00, Cl = 35.5

(iv) A sample in 1.0 cm cell is determined to transmit 45 % of light at a certain wavelength. If the absorptivity of this substance at this wavelength is 2.5 cm^{-1} g^{-1} dm^3, what is the concentration of the substance?

(v) What are the usual X, Y axes on an UV/visible spectrum and an infrared spectrum?

(vi) What is meant by the term resonance absorption in atomic spectroscopy?

(vii) What is the water regain value in size exclusion chromatography?

(viii) Name one common detector in each case, which is suitable for use in gas chromatography and high performance liquid chromatography.

(ix) Distinguish between the terms fundamental, overtone and combination band, with respect to infrared.

(x) List three characteristics of the mobile phase in gas chromatography.

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

(xii) Draw a block diagram of a stablisied d.c. power supply unit.

(xiii) Give the circuit diagram symbol for a L.E.D. and state one use of this device.

(xiv) A lens has a diameter of 6 cm and a focal length of 60 cm.
Calculate the speed number of this lens.
Would this lens be suitable for use in the monochromator of an u.v.- visible spectrophotometer?

Section B

Q2. (a) List the components of the HPLC instrument, and give a one line description of the function it fulfils. (5 marks)

(b) The most common mode of operation in HPLC is reverse phase chromatography.
Explain the term "reverse phase chromatograph" and give an example of a suitable mobile and stationary phase. (4 marks)

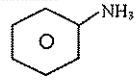
(c) Explain what is meant by bonded phase chromatography.
Give an equation which illustrates how a bonded phase can be formed. (6 marks)

(d) When planning a separation using HPLC a number of fundamental questions must be asked. List these questions. (5 marks)

Q3. (a) Reproduce and complete the following tables in your answer book: (7 marks)

Wavelength nm	*Region of Electromagnetic spectrum*	*Cell Material*	*Common solvent*
10 – 200	-	-	-
-	near ultraviolet	-	-
-	-	glass/plastic	-

(b) Consider the following:

Species	*Λ / nm*	*ε / mol^{-1} dm^3 cm^{-1}*
	255	23
	280	143

Adding a NH_2 group to benzene affects both the wavelength position and the molar absorptivity.

(i) Name and explain briefly the effect NH_2 group has on benzene in this scenario.

(ii) Identify at least <u>one</u> other substituent, which cause similar effects. (6 marks)

(c) σ , π, n, σ *, Π * are all types of molecular orbitals (MO).

(i) Label the MO's as bonding or antibonding.

(ii) Between which orbitals do transitions most often occur? Justify your selection.

(iii) Which transitions require absorption of the most and the least energy to occur.

(iv) Draw a labelled energy level diagram to illustrate the energy ranking between MO.

(7 marks)

Q4. Attempt <u>three</u> of the following:

(i) What mass of $Cr(NO_3)_3 . 9H_2O$ is required to prepare 1.5 dm^3 of 100 ppm chromium stock solution? What is the concentration of this solution in mol dm^3?

Cr = 51.99 a.m.u.; N = 14.00 a.m.u.; O = 16.00 a.m.u.; H = 1.01 a.m.u.

(ii) Describe the mechanism of partition chromatography.
Supplement your answer with a diagram.

(iii) A band of solute becomes broader as it moves through a chromatography column. Identify and describe <u>three</u> reasons why this occurs. Diagrams may aid your description.

(iv) Compare and contrast packed and capillary columns used in gas chromatography.

(v) The mid-infrared region can be divided into four distinct subsections.

Identify three by name and wavenumber range. Which is the most useful?

Q5. (a) Flame atomic emission spectrometry and atomic absorption spectrometry are related atomic spectroscopic techniques.

List three similarities and two differences between the methods. (6 marks)

(b) A sample was analysed for sodium content using flame atomic emission spectrometry. An emission value of 755 was recorded. A series of standards were prepared and their emission values determined. See table below. As can be seen the sample emission is outside the range of the standards emission. The sample was then diluted by taking 10 cm^3 of the original solution and making up to the mark in 50 cm^3 volumetric flask with water. This diluted sample was analysed, its emission value is shown on the data table.

Conc. / ppm	*Emission*
1	25
2.5	75
5	185
10	375
15	575
Sample	755
Diluted sample	255

(i) Plot the appropriate calibration curve.

(ii) Determine the concentration of sodium in the original sample. (10 marks)

(c) The standards listed in the table were prepared by dilution of 150 ppm stock solution. What volume of this stock solution is required to prepare:

(i) 100 cm^3 of the 2.5 ppm standard solution

(ii) 25 cm^3 of the 15 ppm standard solution. (4 marks)

Section C

Q6. (a) (i) Sketch a diagram showing the optical arrangement of a Czerney-Turner monochromator. (4 marks)

(ii) Calculate the resolving power in first order for a grating with 1000 grooves/mm when 3 cm of the grating is illuminated. (4 marks)

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

(b) (i) Draw a circuit diagram of a common emitter voltage amplifier circuit. (5 marks)

(ii) Comment on the biasing arrangement of the transistor.
(Refer to the connections in the circuit diagram you have drawn) (3 marks)

Q7. (a) (i) Describe, with the aid of a diagram, a photomultiplier tube. (6 marks)

(ii) State the properties of an ideal detector. (4 marks)

(iii) Discuss briefly the performance of the photomultiplier tube as an □ideal□detector. (2 marks)

(b) An interference filter is designed using a layer of cryolite (refractive index n = 1.35) between two semi-transparent silver layers. The filter is to transmit the yellow mercury line of wavelength 579 nm (first order) (1 nm = 1 x 10 $^{\square 9}$ nm).

(i) Draw a diagram of the arrangement. (4 marks)

(ii) Calculate the thickness of the cryolite layer needed. (4 marks)