

Cork Institute of Technology

Bachelor of Science in Cell & Molecular Biology- Award

(National Diploma in Science in Cell and Molecular Biology-Award)

Bachelor of Science in Food Science & Technology- Award

(National Diploma in Science in Food Science & Technology-Award)

(NFQ – Level 7)

Autumn 2005

ANALYTICAL CHEMISTRY

(Time: 2 Hours)

Answer any FOUR questions

Examiners: Prof. R. Fitzgerald
Dr. L. Goold

1. The following data refer to the quantitation of an analyte (**A**) in a solid sample by a method of analysis using the method of internal standard (**B**) quantitation. The analyte, A, was quantitatively extracted from 1.650g of the sample into 25.0 cm³ of an appropriate solvent. The internal standard, B, was added to this sample extract solution and all standard solutions of the analyte in such a way that its concentration was the same in all solutions.

<u>DATA:-</u>	A	B	CONC. OF A
	Measurement Value	Measurement Value	(ppm)
	6470	23640	0.50
	10500	20510	1.00
	17470	16140	2.00
	33080	20090	3.00
	13820	20150	sample extract

- (a) Estimate, using an appropriate plot, the concentration of analyte in the original sample in (i) ppm and (ii) %(w/w). (15 marks)
- (b) What are the factors that must be considered when choosing a material as an internal standard in a particular analysis? (5 marks)
- (c) Mention a particular method of analysis where the internal standard method of quantitation is likely to be more appropriate than simple calibration and briefly outline the reason for your choice. (5 marks)

2. (a) State the 4 major forms of chromatography classified according to the nature of the stationary phase. In each case give a brief description of the mechanism by which the components separate based on their interaction with the stationary phase. (10 marks)
- (b) Describe how the following chromatographic parameters can be determined from a chromatogram:
- (i) capacity factor, k^1 ,
 - (ii) theoretical plates, N ,
 - (iii) selectivity factor, α , and
 - (iv) resolution, R .
- What is the relationship between resolution, R , and theoretical plates, N , in a chromatographic process? (15 marks)
3. (a) Describe, using a block diagram, the essential features of a gas chromatographic instrument. Give a brief description of each component that you refer to. (15 marks)
- (b) Discuss the principles of operation of a flame ionisation gas chromatographic detector. Compare its performance with some of the other more commonly used detectors in gas chromatographic analysis. (10 marks)
4. (a) Compare normal and reverse modes of HPLC analysis. (6 marks)
- (b) Write brief explanatory notes on the following aspects of HPLC instrumentation:
- (i) dead volume
 - (ii) pre(guard)-column
 - (iii) gradient programmer. (12 marks)
- (c) Describe the bonding of octadecylsilane(C_{18}) stationary phase on to SiO_2 . (7 marks)
5. Write an essay on uv-visible spectrophotometry using the following headings as guidelines:
- (i) principles
 - (ii) comparison of single and double beam instruments
 - (iii) type of molecule that absorbs uv-visible electromagnetic radiation
 - (iv) qualitative and quantitative analysis.
- (25 marks)