

# Cork Institute of Technology

## Bachelor of Science in Cell & Molecular Biology - Award

## Bachelor of Science in Food Science & Technology - Award

December 2004

### Analytical Chemistry

(Time: 2 Hours)

Answer any FOUR questions

Examiners: Prof. R. O Kennedy  
Dr. L. Goold

- Q1. (a) Outline the steps involved in the solution of a quantitative analytical problem and **briefly** explain the significance of each step (8 marks)
- (b) Describe the sub-sampling process of coning and quartering of solid particulate matter (4 marks)
- (c) Explain how the soxhlet method of liquid-solid extraction operates (8 marks)
- (d) Calculate the mass of KCl required to prepare 100 cm<sup>3</sup> of a 250 ppm solution of K (relative atomic mass data:- K=39.1, Cl=35.5) (5 marks)

- Q2. An analyst determines the concentration of a food additive in a solid food sample by a suitable method of analysis but uses two different calibration methods i.e. simple calibration and standard addition calibration. **0.3200g** of the original sample were dissolved and diluted to a **litre** to give the sample test solution.

**Simple Calibration Procedure**:- A series of standard solutions were prepared by pipetting **1.0, 2.0, 3.0, 4.0** and **5.0** cm<sup>3</sup> aliquots of a standard solution of the analyte (concentration=**50ppm**) into a series of **25.0** cm<sup>3</sup> volumetric flasks and diluting each flask to volume. The measurement values for these solutions were **0.126, 0.250, 0.374, 0.500** and **0.626**, respectively. The sample test solution measured **0.220**.

**Standard Addition Procedure**:- Five **10** cm<sup>3</sup> aliquots of the sample test solution were measured into separate **20.0** cm<sup>3</sup> volumetric flasks. **1.0, 2.0, 3.0, 4.0** and **5.0** cm<sup>3</sup> aliquots of a standard solution of the analyte (concentration=**40ppm**) were added to each of the flasks before each flask was diluted to volume. The measurement values for these solutions were **0.260, 0.383, 0.510, 0.635** and **0.762**, respectively.

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- (a) Construct calibration plots (on separate sheets of graph paper) and , for each plot, determine the concentration value of the analyte in the sample test solution. (16 marks)
- (b) In your opinion, which type of calibration plot gives the most reliable concentration value? Give a reason for your answer. (4 marks)
- (c) Use **either** concentration value, determined in (a) , to calculate the %(w/w) of analyte in the original food sample. (5 marks)

- Q3. (a) Describe the manner in which separation is achieved in the following types of chromatography:- (i) partition (ii) ion-exchange and (iii) size exclusion. (7 marks)
- (b) Explain, using relevant equations, how the following chromatographic parameters can be determined from a chromatogram:- (i) capacity factor of a component,  $k^1$  (ii) column theoretical plate value, N and (iii) resolution, R, between two different components. (6 marks)
- (c) Discuss the following sample introduction techniques in gas chromatographic analysis:- (i) head space analysis and (ii) stream splitting. (6 marks)
- (d) Give two reasons why derivatisation of analyte is sometimes necessary in gas chromatographic analysis. Briefly describe an appropriate derivatisation procedure for (i) sucrose and (ii) a fatty acid. (6 Marks)

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- Q4. (a) Use a labelled block diagram to illustrate the essential components of a HPLC instrument and **briefly** describe the function of each component. (12 marks)
- (b) 'The silanol groups on the surface of silica particles play an important role in the production of suitable stationary phase material for HPLC analysis'. Discuss this statement. (7 marks)
- (c) Explain the meaning of reverse phase HPLC analysis and describe how retention times can be controlled in this type of HPLC analysis. (6 marks)

Q5. Write a detailed essay comparing uv -visible and fluorescence spectroscopy, using the following headings as guidelines:-

- (a) Principles upon which the techniques are based. (5 marks)
- (b) Instrumentation. (10 marks)
- (c) Selectivity. (5 marks)
- (d) Sensitivity (5 marks)

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