CORK INSTITUTE OF TECHNOLOGY
INSTITIÚID TEICNEOLAÍOCHTA CHORCAÍ

Semester 1 2011/12

Module Title: Bio Analytical Science III

Module Code: **CHEA6003**

School: Science & Informatics

Programme Title:
Bachelor of Science in Applied Biosciences & Biotechnology – Year 2
Bachelor of Science (Honours) in Pharmaceutical Biotechnology – Year 2
Bachelor of Science (Honours) in Nutrition & Health Science – Year 2

Programme Code: **SBIOS_7_Y2**
SPHBI_8_Y2
SNHSC_8_Y2

External Examiner(s): **Dr. C. Lennon**
Internal Examiner(s): **Dr. R. Hourihane**
Dr. M. Lehane
Ms. E. Norris

Instructions: **Attempt Three questions.**
Section A, Question1, is compulsory.
Attempt any two questions from Section B
Show all calculations and rough work on the answer script

Duration: 2 Hours
Sitting: Semester 1 2011/12

Requirements for this examination: Periodic Table

Note to Candidates: Please check the Programme Title and the Module Title to ensure that you have received the correct examination paper.
If in doubt please contact an Invigilator.

Section A - COMPULSORY

Q1. Attempt any 8 of the following 12 parts. All carry equal marks.

(i) Give a description of the infra-red, visible and ultraviolet regions of the electromagnetic spectrum. Include detail about common properties and differences between these wavelength regions. What is the wavelength range for the infra-red, visible and ultraviolet parts of the electromagnetic spectrum? [5 marks]

(ii) State three types of electromagnetic spectroscopy. Give a brief description of one of these types of spectroscopy. [5 marks]

(iii) Describe a laboratory experiment that illustrates either (i) characteristics and behaviour of a light detector, or (ii) characteristics of light sources suitable for use in spectroscopy. [5 marks]

(iv) In relation to spectroscopy, what is the function of a monochromator? Draw a diagram that illustrates the optical layout of a Czerny-Turner monochromator. Write down the grating equation. Explain why groove spacing on a grating is important. [5 marks]

(v) Most spectroscopic analysis techniques requires electromagnetic radiation to have narrow bandwidth. Explain what 'narrow bandwidth' means. Why is a narrow bandwidth desirable for spectroscopic applications? List three methods/techniques for achieving narrow bandwidth light source. [5 marks]

(vi) Reflection gratings are an important component in spectroscopic instrumentation. What function does the reflection grating perform? Write down the grating equation and identify each variable. A reflection grating has a groove separation of $7.14 \times 10\text{-}7$ m. Light is incident on the grating at 10^{o} and has an first order angle of diffraction of 20^{o}. Calculate the first order diffracted wavelength of light. [5 marks]

(vii) Calculate the molarity of a solution marked 70% HNO_3, (nitric acid), density = 1.42 gcm^{-3}. Mr. H = 1.00 $gmol^{-1}$, N = 14.00 $gmol^{-1}$, O = 16.00 $gmol^{-1}$

(viii) A 50 cm^3 sample of cough mixture was analysed and found to contain 25mg of glucose. Express the concentration of glucose in pph and ppm.

(ix) A 7.50 x 10^{-3} mol/L solution of potassium permanganate has a transmittance of 36.4 % when measured in a 10mm cell at a wavelength of 525nm. Calculate

(a) the absorbance of this solution

(b) what would be the absorbance of a solution twice as concentrated

(c) calculate the molar absorptivity of $KMn0_4$

(x) Draw a simple, labelled energy level diagram, which describes the process of fluorescence emission. Name the two radiationless emission processes which, compete with fluorescence, include them in the energy level diagram

(xi) Describe using a sketch how the Retention/ retardation factor (Rf) value is calculated for a spot on a thin layer chromatography plate.

(xii) In instrumental analytical methods what is meant by the limit of detection?

(xiii) Describe using a sketch the nature of reverse phase stationary phase used to pack chromatography columns.

(40 marks)

Section B

Attempt any two of the following three questions. All carry equal marks.

Q2.(a) Determination of a metal by ultraviolet visible spectrometry often requires the formation of a metal chelate or complex.

(i) Explain briefly why this is necessary?

(ii) What effect does chelation have on the position and intensity of the lamda max?

(iii) Identify and discuss briefly three important characteristics an ideal chelating reagent must possess. (8 marks)

(b) A sample of atmospheric water was analysed, according to literature methods, for iron content using molecular ultraviolet spectroscopy. To this end, a series of standards were prepared from a 100 ppm iron stock solution. Their absorbance values were measured at 510 nm. The data for both sample and standard is contained in the data table below. As can be seen from the data, the absorbance value for the water sample is outside the range of the standards. The water sample was thus diluted to 10% of its original concentration, and re-analysed.

The absorbance of the diluted sample is shown on the data table also.

(i) Plot the appropriate calibration curve

(ii) Determine the concentration of iron in the **original** water sample.

Absorbance	[Fe] /ppm
0.009	1.00
0.028	3.00
0.048	5.00
0.067	7.00
0.096	10.00
0.900	Water sample
0.081	Diluted water sample

(10 marks)

(c) The standards listed in the table were prepared from 100 ppm iron stock solution. What volume of this stock is required to prepare:

(i) 100 cm^3 of the 7 ppm standard solution

(ii) 25 cm^3 of the 1 ppm standard solution (4 marks)

(d) (i) List three requirements for the application of Beer's law to be successful.

(ii) Identify and explain briefly three deviations from Beers Law. (8 marks)

Q3. (a) What is analytical chemistry? Outline the steps involved in any analytical process. (4 marks)

(b) (i) Sampling is the process of selecting a representative bulk sample from the lot. Sample preparation is the process that converts a bulk sample into a homogenous laboratory sample. Aliquots are then taken for analysis. Sometimes in analysis it is necessary to mask an interfering species. Explain clearly the underlined terms.

(ii) Distinguish between sensitivity and selectivity of a method. Give an example of a method, which is "good" in both. (14 marks)

(c) (i) Illustrate the conductiometric titration graph, (well labelled), obtained when a strong acid, (HCl) is titrated against a strong base, (NaOH).

(ii) Explain how the end point is determined from the graph.

(iii) Write an equation for the overall reaction.

(iv) Identify the ions which, contribute to the conductivity of the solution before, at and after the end point. (12 marks)

Q4. (i) Using a normal phase column and a moderately polar mobile phase predict the order of elution (from 1st to last) of the following analyte components by liquid chromatography (LC): an ester, a carboxylic acid, an alkene, a ketone and an ether.

(ii) Using the terms A, B and C of the Van Deemter Equation explain the reasons for the peak broadening observed in chromatography peaks.

(iii) What measures can an analyst take to improve the peak shape and width when developing a chromatography method?

(iv) Discuss the interactions between the analyte and the mobile and stationary phases in ion chromatography.

(v) Define what are meant by the following chromatographic terms:

a. Retention Time
b. Peak Resolution
c. Height Equivalent to a Theoretical Plate (HETP)
d. Buffer.

(Total 30 marks, 6 marks each)