UNIVERSITY OF SWAZILAND
FIRST SEMESTER EXAMINATION, 2009/2010

TITLE OF PAPER	:	**Special Analytical Techniques**
COURSE CODE	:	**C514**
TIME ALLOWED	:	**Three (3) Hours.**
INSTRUCTIONS	:	**Answer any Four (4) Questions. Each Question Carries 25 Marks**

DO NOT OPEN THIS QUESTION PAPER UNTIL PERMISSION TO DO SO HAS BEEN GRANTED BY THE CHIEF INVIGILATOR.

Question 1 (25 marks)

(a) As briefly as possible, discuss the interactions of the following radiations with matter

(i) β-rays [10]

(ii) γ-rays [15]

Question 2 (25 marks)

(a) The Neutron Activation Analysis, (NAA), is a popular method of activation analysis. For this method (i.e NAA) :

(i) Distinguish between the two types i.e RNAA and INAA. [2]

(ii) Briefly discuss the general principles of this method and the general steps usually taken when employing this method. [5]

(iii) Identify the main sources of neutrons for this method. [3]

(iv) Give three advantages and two limitations of this method [5]

(v) Summarize the procedure for the INAA (instrumentation neutron activation analysis). [4]

(b) A 0.500-g sample of newly developed Ni alloy and 1.00-g of a standard alloy were set up in a nuclear reactor for irradiation with neutrons. On completion of irradiation, both the sample and the standard were allowed to cool. Their activities were found to be 1020 counts/min for the sample and 3540 counts/min for the standard. If the standard was known to contain 5.93% w/w Ni, calculate the % w/w Ni in the new alloy, using the method of external standards. [6]

Question 3 (25 marks)

(a) For the 'Isotope Dilution Analysis' :

(i) Give a general procedure for the method. [2]

(ii) What are the requirements for a successful application of this method? [3]

(iii) Give the specific procedural steps involved when employing the Direct Isotope Dilution analysis for the particular analysis. [4]

(b) Sate the advantages and limitations of the following isotope dilution analytical methods.

(i) Direct Isotope dilution analysis, (DIDA).

(ii) Indirect isotope dilution analysis, (IIDA).

(iii) Radiorelease method of analysis. [9]

(c) On employing the isotope dilution method for the determinations of the concentration of insulin in a sample, a 1.00-mg sample of insulin labelled with ^{14}C, with an activity of 549 counts/min was added to a 10.00ml sample. After adequately homogenizing the sample, a portion of the insulin was separated and purified, giving 18.30 mg of pure insulin. The measured activity of the isolated insulin was 148 counts/min. Calculate the amount of insulin (in mg), present in the original sample. [7]

QUESTION 5 [25]

a) (i) Briefly discuss two reasons why an analytical laboratory with AAS intrumentation may want to carry out liquid-liquid extraction prior to analysis. [4]

(ii) Use diagrams to describe the liquid-liquid extraction procedure for trace element analysis. [2]

(iii) List and discuss any two (2) major disadvantages associated with liquid-liquid extractions in the analytical laboratory. [4]

b) (i) An analytical laboratory routinely extracts Ni from industrial waste water as the isocyanate prior to AAS analysis using an FIA-AAS system. What does the acronym " FIA-AAS" stand for? [1]

(ii) State the difference between "batch extraction" and "continuous extraction" in analytical chemistry. [2]

(iii) Draw and label the FIA system used for the Ni extraction. [4]

(iv) In the system described in b (iii) above, explain the reason for fragmenting the solvent into a bolus flow. [2]

(v) Use drawings to explain how the sample loop injection valve for introducing the waste water sample works. [3]

(vi) How is the Ni^{2+} quantified in an FIA-AAS instrument? [3]

QUESTION 6 [25]

a) (i) Outline and describe the major steps involved in solid phase extraction (SPE) prior to analysis of Aflatoxin A in peanuts. [2]

(ii) Use diagrams to describe the SPE mode "digital chromatography" [2]

b) (i) Describe the role of "stream splitting" in LC-MS. [2]

(ii) Describe the method of electrospray ionization in LC-MS. [2]

(ii) Explain how the quadrupole unit acts as a detector in LC-MS. [2]

c) (i) Give an estimate of the temperatures attainable by ICP, and explain how this makes an ICP a good ion source for mass spectrometry. [2]

(ii) Outline the major challenge of interfacing an ICP instrument to a quadrupole mass spectrometer. [2]

(iii) Use diagrams to explain how the interface between an ICP and a quadrupole mass spectrometer works. [3]

(iv) List and describe two advantages of ICP-MS over ICP-OES. [4]

(v) List and describe two interferences in ICP-MS. [4]