

**UNIVERSITY OF SWAZILAND**  
**FIRST SEMESTER EXAMINATION, 2010/2011**

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**TITLE OF PAPER** : **Special Analytical Techniques**

**COURSE CODE** : **C614**

**TIME ALLOWED** : **Three (3) Hours.**

**INSTRUCTIONS** : **Answer any Four (4) Questions. Each Question Carries 25 Marks**

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

**Question 1 (25 marks)**

- (a) For the  $\beta$  - particle, briefly discuss the following:
- (i) The forms of decay processes, giving an illustrative example in each case. [5]
  - (ii) The nature/types and effects of its interaction with matter. [6]
- (b) Discuss the types and effects of the interaction of  $\gamma$ -radiation with matter. [6]
- (c) A sample containing  $N_0$  atoms of a given radioactive material, X, with  $t_{1/2} = 37.30$  minutes was received at 14.00 hrs for  $\beta$  - counting. It was placed in a liquid scintillation counter with 100 % efficiency at 16.30 hrs and counted for 60.00 minutes. If the accumulated counts were 100,000 calculate  $N_0$  [8]

**Question 2 (25 marks)**

- (a) For the 'Gas — Ionization Detectors':
- (i) Briefly describe their working principles. [3]
  - (ii) On the basis of the nature of the filler gas and the applied bias voltage, identify the main types of ionization detectors that emerge in this class. Use an accompanying diagram/figure where necessary. [12]
- (b) Give two other classes of commonly employed nuclear radiation detectors. [2]
- (c) In an experiment where the isotope dilution analysis was employed for the determination of the amount of Co in steel, a 1.00-g sample of steel was dissolved in an acid and exactly 2.00 mL of a spike solution of  $^{60}\text{Co}$  was added to the solution. The concentration of the spike solution was 3.00 mg/mL of Co, with an activity of  $1.50 \times 10^4$  dpm per mg of Co. The  $\text{Co}_2\text{O}_3$  plated out on an anode immersed in the solution increased its weight by 12.50 mg and its activity was  $2.5 \times 10^3$  dpm. Calculate the w/w % of Co in the steel sample. [8]

**Question 3 (25 marks)**

- (a) The Neutron Activation analysis, (NAA), was a popular method of activation analysis. For this method (i.e NAA):
- (i) Distinguish between the two was 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 (Instrumental neutron activation analysis). [4]

(b) A 0.500-g sample of a newly developed Ni alloy and a 1.000-g of a standard alloy were set up in a nuclear reactor for irradiation with neutrons. On completion of the 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. [5]

#### QUESTION 4 [25]

- a) Use diagrams to explain how a photodiode array (PDA) works as a detector in UV-visible spectroscopy, and briefly discuss its advantages over other alternative detectors in regard to detection limits. [5]
- b) Given that the signals from a photodiode array detector are in the form of current, which must be converted to voltage for input into a computer for electronic display, draw the operational amplifier that must be used at this stage of signal processing, and state its output. [4]
- c) Given that the signal from (b) above will have a noise component to it, draw an electronic circuit that will filter out the noise. [3]
- d) Given that the noise-free signals from the reference and sample channels in the PDA must be converted to transmittance, draw the operational amplifier that must be used at this stage of signal processing, and state its output. [4]
- e) Given that the transmittance must be converted to absorbance, draw the operational amplifiers that must be sequentially added, and state their respective outputs. [6]
- f) State the "Nyquist Theorem", and explain how is it applied when sampling the analog signals from (e) above for analog-to-digital conversion. [3]

#### QUESTION 5 [25]

- a) (i) State the difference between "batch extraction" and "continuous extraction" in analytical chemistry. [2]
- (ii) State two reasons why liquid-liquid extraction of copper as the cupferon complex is carried out in rock samples prior to analysis. [2]
- (iii) Use diagrams to describe how the liquid-liquid extraction procedure for copper as the cupferon complex is carried out. [3]

- b) Solid Phase Extraction (SPE) is now a preferred method for prior to analysis of mycotoxins in food grains such as maize.
- (i) Outline and describe the major steps involved in solid phase extraction. [3]
  - (ii) Use diagrams to describe the SPE mode "digital chromatography" [3]
- c) Flow injection analysis with atomic absorption detection is one of many hyphenated analytical techniques in use today.
- (i) Draw and label an FIA system. [4]
  - (ii) Explain the reason for fragmenting the solvent into a bolus flow in flow injection analysis. [2]
  - (iii) Use drawings to explain how the sample loop injection valve for introducing samples works. [3]
  - (iv) How is quantification carried out in an FIA-AAS instrument? [3]

**QUESTION 6 [25]**

- a) LC-MS is a one of several hyphenated analytical techniques in use for measurement of organic compounds in liquid samples.
- (i) Describe the role of "stream splitting" in LC-MS. [2]
  - (ii) Describe the method of electrospray ionization in LC-MS. [3]
  - (iii) Explain how a Time of Flight (ToF) units acts as a detector in LC-MS. [4]
- b) GC-MS is one of several hyphenated analytical techniques in use today.
- (i) Explain how ionization of eluents from the column in GC-MS are carried out. [3]
  - (ii) What are the advantages of GC-MS over conventional GC with thermal conductivity detection? [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) Describe the mechanical interface between the plasma and a quadrupole in ICP-MS. [2]
  - (iii) What are the advantages of ICP-MS over ICP-OES. [3]
  - (iv) Using arsenic (As) as an example, explain the causes of isobaric interferences in ICP-MS. [4]