DEPARTMENT OF CHEMISTRY

UNIVERSITY OF SWAZILAND

C614/CHE 611 **SPECIAL ANALYTICAL TECHNIQUES**

DECEMBER 2017 **FINAL EXAMINATION**

Time Allowed: **Three (3) Hours**

Instructions:

1. **This examination has six (6) questions.**
2. **Answer any four (4) questions fully; diagrams should be clear, large and properly labelled. Marks will be deducted for improper units and lack of procedural steps in calculations.**
3. **Each question is worth 25 marks.**

Special Requirements

NONE

YOU ARE NOT SUPPOSED TO OPEN THIS PAPER UNTIL PERMISSION TO DO SO HAS BEEN GIVEN BY THE CHIEF INVIGILATOR.

QUESTION 1 [25]

(a) Explain why it is desirable to carry out a flow injection analysis (FIA) for the atomic absorption spectrochemical analysis of trace Cd in human urine, rather than direct aspiration into the AAS. (1)

(b) (i) Discuss two desirable properties of dithizone as extraction agent in the determination of Cd in human urine by FIA. (2)

(ii) Use diagrams to explain the origins of the Nernst diffusion film in the FIA technique. (3)

(iii) Use diagrams to explain how film wetting results in bandbroadening in FIA. (3)

(c) Describe, using diagrams, each of the following components of an FIA system, and explain their role in the determination of Cd in urine by FIA/AAS

(i) Peristaltic pump (3)

(ii) Segmentor (3)

(iii) Extraction coil (3)

(iv) Phase separator (3)

(d) Use diagrams to explain how Cd in urine is quantified using FIA/AAS. (4)

QUESTION 2 [25]

(a) State the neutron activation equation used to quantify analytes by Instrumental Neutron Activation Analysis (INAA), and explain all terms appearing in it. (4)

(b) Using ^{59}Co in a soil sample as an example, use nuclear equations to explain the nuclear capture process, the generation of radioactive isotopes, and the emission of gamma rays which are used for quantification in INAA (4)

(c) (i) Draw a schematic of a nuclear reactor using uranium rods to generate neutrons in INAA (4)

(ii) In this reactor, explain the role of the:

- Cd rod (2)
- Be shield (2)
- water pool (2)

(d) (i) Explain how samples are introduced to the reactor in INAA (2)

(ii) Explain how gamma ray spectroscopy is used to quantify Co in a soil sample by INAA (3)

(iii) Why would an analyst prefer to use INAA to determine elements in soil samples over the more conventional flame atomic absorption spectrometer (2)

QUESTION 3 [25]

(a) Use diagrams to explain how a

(i) Single Focus Time of Flight Mass Spectrometer works (4)

(ii) Double Focus Time of Flight Mass Spectrometer works (4)

(b) Describe the purge-and-trap GC-MS technique (4)

(c) What is meant by tandem GC-MS? (3)

(d) Use diagrams to explain how electron ionization is achieved in this technique and equations to explain its mechanism. (4)

(e) Use a chemical equation to explain how chemical ionization is achieved in GC-MS. (4)

(f) Explain why an analyst would opt to use GC-MS over the more conventional GC instrument with Thermal Conductivity Detector (TCD), in environmental monitoring. (2)

QUESTION 4 [25]

(a) Use diagrams to explain how a Quadrupole Mass Spectrometer works (4)

(b) Use diagrams to explain how

(i) Electro spray ionization is achieved in LC-MS, using equations to explain its mechanism (4)

(ii) Fast atom bombardment is achieved in LC-MS, using equations to explain its mechanism. (4)

(c) Describe each of three steps used in Matrix-Assisted Laser Deposition/Ionization (MALDI) used in LC-MS. (6)

(d) Use diagrams to describe the direct electron ionization LC-MS interface (4)

(e) Explain why an analyst would opt to use LC-MS over the more conventional HPLC technique with UV-visible detection, in drug analysis (3)

QUESTION 5 [25]

(a) Use energy level diagrams and Planck's Equation to explain principles of x-ray fluorescence. (3)

(b) Use diagrams to explain how conventional x-ray generators work in XRF. (3)

(c) Describe how soil samples are prepared for XRF analysis. (2)

(d) Explain how:

(i) Geiger counters (3)

(ii) Scintillation counters (3)

work in detecting incoming radiation in wavelength dispersive XRF

(e) Describe how the following interferences affect XRF analysis

(i) X-ray absorption (3)
(ii) X-ray enhancement (3)
(iii) Sample macroscopic effects (3)

(f) Why would analyst opt to use XRF for elemental analysis of soil over ICP optical emission techniques? (2)

QUESTION 6 [25]

(a) Explain the fundamental principles of Scanning Electron Microscopy (SEM). (3)

(b) Draw the schematic of an SEM, and explain how the electron beam is produced. (3)

(c) Use diagrams to explain the mechanisms of the emission of three signal types during a raster scan in SEM. (3)

(d) Describe how non-conductive samples such as those containing asbestos fibres are prepared in SEM (2)

(e) Explain how it is possible to carry out energy dispersive analysis with SEM. (3)

(f) (i) Explain how incoming photons are detected and processed by solid state detectors in energy dispersive analysis. (3)

(ii) Explain how incoming photons are detected and processed by proportional counters in energy dispersive analysis. (3)

(iii) Explain how incoming photons are detected and processed by PIN diode in energy dispersive analysis.(3)

(g) Why would an anlyst opt to use SEM with EDS when monitoring mining and rehabilitation at asbestos mines? (2)