

DEPARTMENT OF CHEMISTRY  
UNIVERSITY OF ESWATINI

C614/CHE 611

SPECIAL ANALYTICAL TECHNIQUES

NOVEMBER 2018

FINAL EXAMINATION

Time Allowed:

Three (3) Hours

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**Instructions:**

1. This examination has six (6) questions.
2. Answer question 1 and any other three (3) questions; 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) Use diagrams to explain the difference between "batch extraction" and "continuous extraction" in analytical chemistry. (3)
- (b) Metal chloro complex  $MCl_3$  is extremely soluble in ether, with the distribution coefficient for a water/ether system being 50. Calculate the concentration of  $MCl_3$  left in 50ml of aqueous 0.01M  $FeCl_3$  solution after extraction
- (i) once with a 10-mL portion of ether (2)
  - (ii) once with a 20--mL portion of ether (2)
  - (iii) twice with 10mL portions of ether (2)
- (c) Describe two ways of recovering analytes from an organic solvent during the stripping stage of solvent extraction. (4)
- (d) 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)

**QUESTION 2 [25]**

- a. The inductively coupled plasma, coupled to a mass spectrometer, is one of the most useful hyphenated techniques in use today.
- (i) Use diagrams to explain how the excitation temperatures of the ICP are measured. (4)
  - (ii) What excellent characteristics make the ICP an ideal source for analytical mass spectrometry? (2)
- b. There are certain operational difficulties associated with coupling a quadrupole unit to an ICP.
- (i) Draw a schematic diagram of an ICP-MS instrument that uses a quadrupole unit, and explain how ions from the ICP are separated in the quadrupole unit. (4)
  - (ii) Use diagrams to explain how the interface in an ICP-MS instrument works. (3)
  - (iii) How are the ions coming out of the quadrupole detected? (3)
- c. Discuss each of the following interferences in ICP-MS
- i) Isobaric interferences (3)
  - ii) Polyatomic interferences (3)
  - iii) Doubly charged ion interferences (3)

**QUESTION 3 [25]**

- (a) Use a diagram to explain why a double focusing magnetic analyzer is superior over a single focusing instrument in GC-MS. (3)
- (b) The interface is very important in coupling a GC to a mass spectrometer.
- (i) Use a diagram to explain how direct coupling works, and describe the most appropriate columns for this type of interfacing. (4)
  - (ii) Use a diagram to explain how a jet separator works in GC-MS. (3)
  - (iii) Use a diagram to explain how an effusion separator works in GC-MS (3)
- (c) Ionization of analytes from a gas chromatograph is one of the most essential steps prior to introduction into a mass analyzer.
- (i) Use a diagram to explain how electron ionization is achieved in GC-MS. (3)
  - (ii) Use a diagram to explain how chemical ionization is achieved in GC-MS. (3)
- (d) Discuss the chemistry involved, and apparatus used in the generation of Hg in the cold vapour atomic absorption spectroscopic technique. (6)

**QUESTION 4 [25]**

- (a) Flow injection analysis with atomic absorption detection is a hyphenated analytical technique used to measure hexavalent chromium in waste water.
- (i) Draw and label an FIA system. (5)
  - (ii) Use diagrams to describe the concept of the "Nernst Diffusion Layer" in flow injection analysis. (4)
  - (iii) Use drawings to explain how a phase separator works in flow injection analysis. (4)
  - (iv) How is quantification of Cr (VI) carried out in an FIA-AAS instrument? (4)
- (b) In HPLC-MS,
- (i) Explain how flow splitting is achieved. (3)
  - (ii) Use a diagram to explain how electrospray ionization is achieved. (3)
- (c) One of the most sensitive technologies for atomic absorption determination of mercury (Hg) is Cold Vapour – AAS. Explain why flame atomic absorption suffers poor detection limits for Hg. (2)

**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) Explain the fundamental principles of Scanning Electron Microscopy. (3)<sup>4</sup>
- (f) Draw the schematic of an SEM, and explain how the electron beam is produced. (3)
- (i) Use diagrams to explain the mechanisms of the emission of three signal types during a raster scan in SEM.(3)
- (ii) Describe how non-conductive samples such as those containing asbestos fibres are prepared in SEM (2)

**QUESTION 6 [25]**

- (a) Outline the principles involved in Instrumental Neutron Activation Analysis using <sup>59</sup>Co as an example. (4)
- (b) Describe the following as it applies to INAA
- (i) NaI (TI) scintillation detector (4)
- (ii) Semiconductor Ge (Li) detector(4)
- (c) Describe the reactor core in a research type SLOWPOKE nuclear reactor as a neutron source, and how it is moderated and cooled. (4)
- (d) Explain how samples are introduced into the reactor core (3)
- (e) Explain how quantification of analytes is carried out in INAA (4)
- (f) Why would INAA be preferred over atomic absorption methods (2)

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