Uva Wellassa University of Sri Lanka
Faculty of Science and Technology
Department of Science and Technology
300 level 2nd Semester Examination – Jan 2019
MRT 362-3 Analytical Techniques and Instrumentation

Uva Wellassa University

Instructions to candidates
Duration: 03 hours
Number of questions: 6 Essay Questions
Mark allocation: 300 marks
Answer all questions.
Scientific calculators are allowed.

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1.

i. Write down the Bragg's law and derive the equation using a suitable schematic diagram.

(08 marks)

ii. The 'd spacing' of 020 plane of a cubic latice is 2.7 Å.

a. Determine the peak position related to this plane. X ray wavelength is 1.5406 Å.

b. Determine the lattice parameter a.

(16 marks)

iii. Systematic absences of *hkl* planes will decide the centring type. How do you distinguish four centring types depending on the observed peaks corresponding to *hkl* planes.

(08 marks)

iv. In the NaCl cubic lattice, a sodium atom appears at the fractional cordinate (1/2,1/2,0) while a chlorine atom at (1/2,1/2,1/2) . Calculate the interatomic distance. Lattice parameter a = 5.4 Å.

(08 marks)

v. In addition to elemental peaks, other peaks can also be appeared in the XRF spectra. Name five such other peak types.

(09 marks)

2.

i. Write down the Broglie's equation which explains a possibility to enhance the resolution of an electron microscope image and define the terms.

(10 marks)

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ii. Compare the optical microscope and electron microscope.

(10 marks)

iii. Explain the edge effect in scanning electron microscope (SEM) using a suitsble diagram.

(10 marks)

iv. Discuss three major differences between scanning electron microscope (SEM) and transmission electron microscope (TEM).

(12 marks)

v. Explain the sample preparation method for solid sheets to take a transmission electron microscope (TEM) image.

(08 marks)

3.

i. What do you meant by "chromatography"? write a brief description

(10 marks)

ii. Write the definitions for the following terms

a. Analyte
b. Eluate
c. mobile phase
d. stationary phase
e. retention time

(10 marks)

iii. Write down the types of chromatographic techniques according to bed shape and state of mobile phases by giving one example for each.

(05 marks)

iv. What do you meant by "height equivalent of a theoretical plate"?

(05 marks)

v. Van-Deemter equation is an equation which has a relationship between height equivalent of a theoretical plate with flow rate as shown below.

$H = A + B/\mu + C\,\mu$

Where, A = constant representing eddy diffusion & mobile phase mass transfer, B = constant representing longitudinal diffusion, C = constant representing stagnant mobile phase and stationary phase mass transfer

What is the optimized value for best height equivalent of a theoretical plate?

(05 marks)

vi. Draw a sketch diagram of Gas chromatography–mass spectrometry (GC-MS) (Label the components of the instrumentation)

(15 marks)

4.

i. What are the major relevant series of radiations for nuclei energy, electronic transition energy, molecular vibrational energy and molecular rotational energy?

(05 marks)

ii. Write down the Beer Lambert law and explain all the terms

(03 marks)

iii. Calculate the molar absorptivity of a 5.00×10^{-3} mol dm^{3} solution, which has absorb 90 % of incident light, when the path length is 30 mm.

(07 marks)

iv. Briefly explain about Fourier Transform Infrared (FTIR).

(05 marks)

v. Draw a sketch diagram for FTIR instrument.

(10 marks)

vi. Consider the Following reactions and relevant standard reduction potentials.

$A2+ (aq) + e \rightleftharpoons A+ (aq)$ (- 0.50 V)

$D+ (aq) + e \longrightarrow D (aq)$ (0.20 V)

$D (aq) \longrightarrow E (S)$

A, D and E indicate unknown chemical species (not a standard notations). Assume that the product, D is not observed in any further electrochemical reactions.

a. What are three electrodes in a standard CV experiment employs?

(03 marks)

b. Sketch a cyclic voltammogram carried out in electrolyte solution contained A^{2+} (aq)

(07 marks)

c. Sketch a cyclic voltammogram for electrolyte solution contained D^{+} (aq)

(10 marks)

(Take scan range from -1.00 V to +1.00 V with respect to standard hydrogen electrode (SHE). Assume that reactions are taken place as mentioned in above equations and no any other reactions take place at this potential range)

5.

i. What is the function of Differential Scanning Calorimetry?

(06 marks)

ii. Explain the principle of Differential Scanning Calorimetry?

(12 marks)

iii. Briefly explain the three (03) types of Thermogravimetry (TG) analysis.

(12 marks)

iv. State five (05) types of measurements that can be taken by Differential Scanning Calorimetry.

(10 marks)

v. Consider a composite X, which is subjected to Thermogravimetry (TG) analysis, construct a Thermogravimetric curve by given information.

a. 100 - 120 °C - dehydration and wight loss is 15%
b. 360 - 400 °C - intermiadiate decomposition and wight loss 30 %
c. 480 - 500 °C surface oxidation at wight gain is 10%
d. 560 - 600 °C melting of composite
e. 720 - 780 °C Final decomposition at and wight loss 60%

(09 marks)

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6.

i. Explain the function of the three (03) electrode system in Cyclic Voltammetry.

(12 marks)

ii. Consider the Zn-Cu redox reaction. Under standard conditions, what is the cell potential after 1 minute? (Assume that after one minute, $[Cu^{2+}]$ = 0.07 M while $[Zn^{2+}]$ = 1.89 M, E*pc* = +1.31 V and E*pa* = +0.97 V).

Hint – R = 8.315 J K^{-1} mol^{-1}, F = 96,485 C mol^{-1}, 0 °C = 273 K

Nernst equation $E = E^0 - \frac{RT}{nF} - \ln \frac{[Red]}{[Ox]}$

(12 marks)

iii. Describe the atomization techniques used in following devices

a. Lamina Flow Burner

b. Graphite tube

c. Inductively coupled plasma torch

(12 marks)

iv. What are the technical differences between Atomic absorption spectroscopy (AAS) and Atomic emission spectroscopy (AES)?

(14 marks)

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