

THE PAPUA NEW GUINEA UNIVERSITY OF TECHNOLOGY

FIRST SEMESTER EXAMINATIONS – 2020

CH 311 – INSTRUMENTAL ANALYSIS I

FRIDAY 23<sup>rd</sup> JUNE 2020 – 12:50 PM

TIME ALLOWED: 2 HOURS

**INFORMATION FOR CANDIDATES: -**

1. You will have 10 minutes to read the question paper. You **MUST NOT** begin writing in the answer book during this time.
2. **ANSWER ALL QUESTIONS.**
3. All answers **MUST** be written on the answer book provided
4. Calculators are permitted in the examination room. Lecture notes, notebooks plain papers and textbooks are **NOT** allowed.
5. Mobile phones are not allowed. **SWITCH OFF THE MOBILE PHONES.**
6. Show all workings and calculations in the answer book.
7. **DRAW** the **STRUCTURES** clear and visible.
8. **DO NOT** over write.
9. Write your name and student **ID number** clearly on the front page of the answer book. **DO IT NOW.**

**MARKING SCHEME: TOTAL 60 MARKS**

1. (a) Explain the following as related to solvent extraction:
- (i) Partitioning.
  - (ii) Distribution coefficient. [2 marks]
- (b) Acid/base extraction is based on acid/base reactions. Show the general reaction equations between:
- (i) organic acid reacting with NaOH and converted back to acid.
  - (ii) organic base reacting with HCl and converted back to base. [4 marks]
- (c) Draw a block diagram of a CO<sub>2</sub> supercritical solvent extractor. [6 marks]
- (d) In food industries supercritical CO<sub>2</sub> is mainly used and small amount of ethanol is spiked into the extraction vessel as co-extractor. What is the purpose to using ethanol? [2 marks]
- (e) The questions below relate to the class practical on the synthesis and separation of ortho- and para-nitroanilines.
- (i) Why was aniline reacted with acetic anhydride?
  - (ii) In nitrating acetanilide, what was the purpose for adding H<sub>2</sub>SO<sub>4</sub>?
  - (iii) Why were the nitrated acetanilide added to distilled water?
  - (iv) Why wasn't aniline directly nitrated?
  - (v) Why were the ortho- and para- isomers the major products? [5 marks]

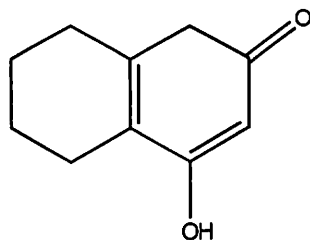
**(Total = 19 Marks)**

2. (a) (i) Name the four types of interactive forces that cause adsorption of solutes onto a chromatographic stationary phase. [2 marks]
- (ii) Explain FOUR reasons for performing Thin Layer Chromatography (TLC) and Paper Chromatography. [4 marks]
- (iii) In preparative Thick Layer Chromatography, what would be the appropriate detector for detecting colourless analytes and explain why? [2 marks]

- (b) Paper electrophoresis of a mixture of lysine ( $pI = 9.47$ ), histidine ( $pI = 7.64$ ) and cysteine ( $pI = 5.02$ ) is carried out at  $pH 5.02$ . Describe the behavior of each of these amino acids. [3 marks]
- (c) Draw a block diagram of a gas chromatography instrumentation and explain the function of each component. [10 marks]

**(Total = 21 Marks)**

3. (a) (i) Explain the type of energy transitions that give rise to Infra-Red (IR) spectroscopy. [2 marks]
- (ii) What type of information about organic molecules is given by an infrared spectrum? [1 mark]
- (iii) Explain briefly how a IR spectrum can be used to identify an unknown compound. [2 marks]
- (b) Use Woodward's rule to predict  $\lambda_{max}$  for the structure below. [5 marks]



- (c) Using the attached spectral data;
- (i) draw a suitable structure.
- (ii) briefly explain how each spectra (IR, UV, Mass Spectra,  $^{13}C$  &  $^1H$ ) is consistent with the structure. [10 marks]

**(Total = 20 marks)**