THE PAPUA NEW GUINEA UNIVERSITY OF TECHNOLOGY

FIRST SEMESTER EXAMINATION

CH313 – INSTRUMENTAL ANALYSIS

TUESDAY 7TH JUNE 2022 8:20 AM

**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** overwrite.
9. Write your name and number clearly on the front page. **DO IT NOW.**

**MARKING SCHEME:** Total 50 marks

1. (a) Indicate whether the following vibrations will be active or not in the Infrared (IR) spectrum.

| Molecule | Motion |
|---|---|
| $SO_2$ | Symmetric stretch |
| $CH_3$-$CH_3$ | C-C stretching |

[2 marks]

(b) The fraction of non-reflected light that is transmitted through a 200 mm thickness of glass is 0.98. Calculate the absorption coefficient of this material.

[4 marks]

(c) Give FOUR reasons, why tetramethylsilane (TMS) is selected as a reference compound in the $^1H$ NMR spectroscopy.

[4 marks]

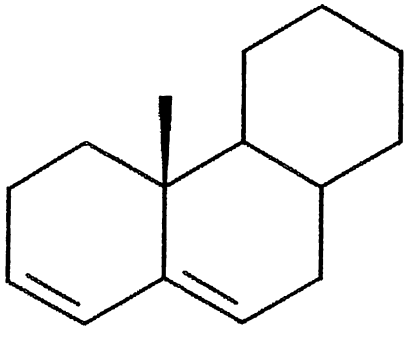
**(Total = 10 marks)**

2. (a) Draw a simple schematic diagram of a gas chromatography (GC) instrument and label its parts.

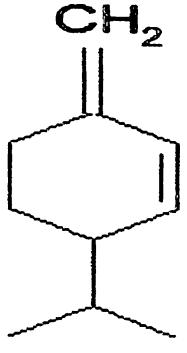
(b) Give any FOUR applications of IR spectroscopy.

(c) Using Woodward - Fieser's rule, calculate the $\lambda_{max}$ for each of the following compounds.

(i)



(ii)




(d) Visualization of the spots in paper chromatography can be done in two ways. What are they? Explain in detail.

(e) Consider the following situation:
There are three components (A, B, C) that need to be separated with each one having different properties. Suggest which one will elute first/ second/ third from a **polar** Gas Chromatography (GC) column (stationary phase) to the detector and explain why?

A polar, least volatile
B non-polar, highly volatile
C polar, highly volatile

**(20 marks)**

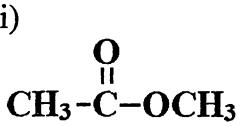
3. (a) Calculate the approximate wave number and wavelength of the fundamental absorption peak due to the stretching vibrations of a carbonyl group. The force constant for a double bond has an approximate value of $1 \times 10^6$ dynes/cm. The masses of carbon and oxygen atoms are $1.8 \times 10^{-23}$ and $2.4 \times 10^{-23}$ g per atom.

(b) Distinguish between Normal-phase HPLC and Reverse-phase HPLC.

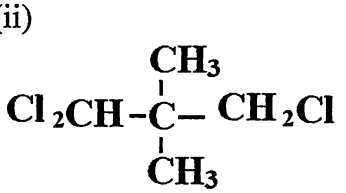
(c) A solution that was $8.14 \times 10^{-3}$ M in Y had a transmittance of 0.234 when measured in a 2 cm cell. What concentration of Y would be required for the transmittance to be increased by a factor of 4 when a 1 cm cell was used?

(d) Using the data in Table 1 (provided in the datasheet), describe the expected $^1$H NMR spectrum of the following compounds.

(i)



(ii)



**(20 marks)**

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# DATA SHEET

## Table 1. Typical $^1H$ chemical shifts (relative to tetramethylsilane)

| Type of $^1H$ | δ (ppm) | Type of $^1H$ | δ (ppm) |
|---|---|---|---|
| C—$CH_3$ | 0.85–0.95 | —$CH_2$—F | 4.3–4.4 |
| C—$CH_2$—C | 1.20–1.35 | —$CH_2$—Br | 3.4–3.6 |
| | | —$CH_2$—I | 3.1–3.3 |
| C—CH—C | 1.40–1.65 | $CH_2$=C | 4.6–5.0 |
| C—C=C | 1.6–1.9 | —CH=C | 5.2–5.7 |
| C—Ar | 2.2–2.5 | Ar—H | 6.6–8.0 |
| —C(=O)— | 2.1–2.6 | —C≡C—H | 2.4–2.7 |
| C—N | 2.1–3.0 | —C(=O)—H | 9.5–9.7 |
| C—O— | 3.5–3.8 | —C(=O)—OH | 10–13 |
| $CH_2$—Cl | 3.6–3.8 | R—OH | 0.5–5.5 |
| $CH_2$Cl | 5.8–5.9 | Ar—OH | 4–8 |

## Table 2. Parent values and increments for different substituents / Groups:

(i) Base value for homoannular diene = 253 nm

(ii) Base value for heteroannular diene = 214 nm

(iii) Base value for acylic diene = 214 nm

(iv) Alkyl substituent or Ring residue attached to the parent diene = 5 nm

(v) Double bond extending conjugation = 30 nm

(vi) Exocyclic double bonds = 5 nm

## Conversion table and physical constants

Speed of light (*in vacuo*), $c = 3 \times 10^8$ m/s

1 Å = $10^{-10}$ m

1 μm = $10^{-6}$ m