



**NAMIBIA UNIVERSITY
OF SCIENCE AND TECHNOLOGY**

FACULTY OF HEALTH, APPLIED SCIENCES AND NATURAL RESOURCES

DEPARTMENT OF NATURAL AND APPLIED SCIENCES

QUALIFICATION: BACHELOR OF SCIENCE	
QUALIFICATION CODE: 07BOSC	LEVEL: 7
COURSE CODE: MSC701S	COURSE NAME: MOLECULAR SPECTROSCOPY AND CHEMICAL SEPARATION METHODS
SESSION: JULY 2022	PAPER: THEORY
DURATION: 3 HOURS	MARKS: 100

SUPPLEMENTARY/SECOND OPPORTUNITY EXAMINATION QUESTION PAPER	
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INSTRUCTIONS
<ol style="list-style-type: none">1. Answer ALL the questions in the answer book provided.2. Write and number your answers clearly.3. All written work MUST be done in blue or black ink.

PERMISSIBLE MATERIALS

Non-programmable calculators

ATTACHMENTS

List of useful formulas and constants

THIS QUESTION PAPER CONSISTS OF 8 PAGES (Including this front page and attachments)

Question 1

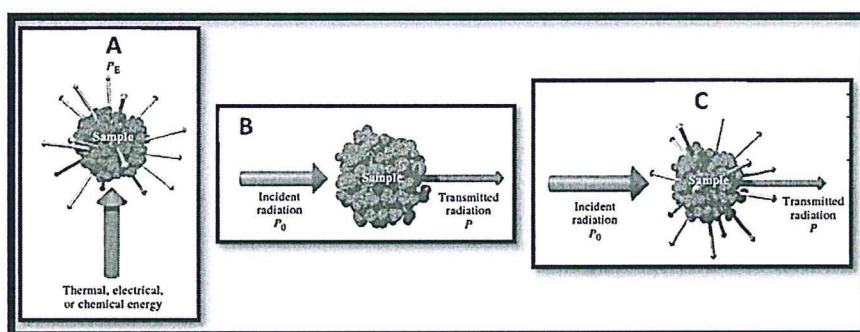
[25]

1.1 Define the following terms

- (a) Sensors (2)
- (b) A readout device (2)
- (c) Absorbance (2)

1.2 It is critical in UV-Vis to measure the 100% transmittance (100%T). This measurement is always carried out with a sample blank. Provide a clear explanation of the relevance of 100%T in UV-Vis and the reason why a blank is used for that measurement. (5)

1.3 The following diagram describes different phenomena that occur when the electromagnetic radiation interacts with the matter



Name the phenomena described in A to C and explain your choices. (6)

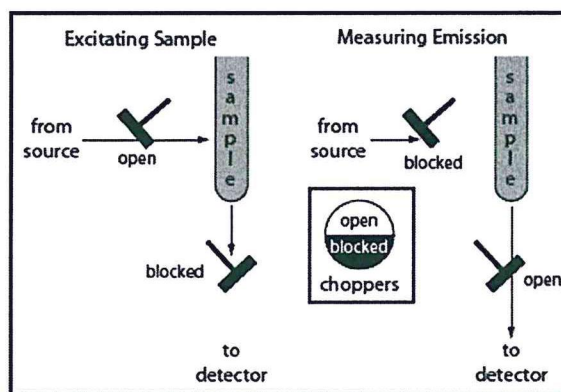
1.4 Differentiate between

- (a) Continuum and line sources of electromagnetic radiation. (4)
- (b) Monochromator and polychromator. (4)

Question 2

[25]

2.1 The following diagram represents a spectrophotometer used for measuring Phosphorescence:



Briefly explain how it operates. (5)

2.2 The following questions are related to the use of plasmas as thermal sources of energy

(a) Use the Boltzmann distribution equation to explain why a plasma provide a better atomization as compared to both flame and graphite furnace. (5)

(b) Besides improving excitation and ionization efficiencies, what other advantage does the higher temperature of plasmas have over flames and furnaces? (2)

(c) Name three types of plasma sources used in optical emission techniques. (3)

2.3 The burner assemblies of atomic absorption spectrometers (AAS) are known to provide a long optical pathlength as well as a stable flame and they can also move horizontally and vertically. Explain the importance of the underlined properties in the statement above. (4)

2.4 The table below shows atomic emission lines for a Cr hollow cathode lamp.

λ (nm)	Slit width (nm)	mg Cr/L giving $A = 0.20$	P_0 (relative)
357.9	0.2	2.5	40
425.4	0.2	12	85
429.0	0.5	20	100
520.5	0.2	1500	15
520.8	0.2	500	20

(a) Which analytical technique uses hollow cathode lamps and why? (2)

(b) For the Cr hollow cathode lamp in the table above, which wavelength provides the best sensitivity? Explain your answer. (2)

(c) When analyzing samples containing ≈ 10 mg Cr/L, which wavelength(s) would you expect to provide a reasonable sensitivity with less uncertainty in the measured

absorbance? Explain your choice.

(2)

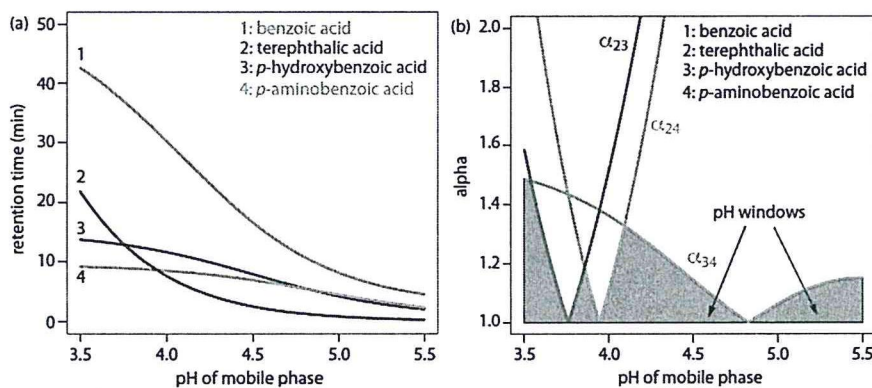
Question 3

[25]

3.1 How do you adjust the selectivity, α , in gas chromatography (GC) and in liquid chromatography (LC)?

(4)

3.2 The figures below show how the mobile phase pH affect both (a) the retention time of the solutes, t_r , and (b) their selectivity, α .



(a) What unwanted situations would occur if the analysis was carried using a mobile phase

(i) at pH between 5.0 and 5.5?

(1)

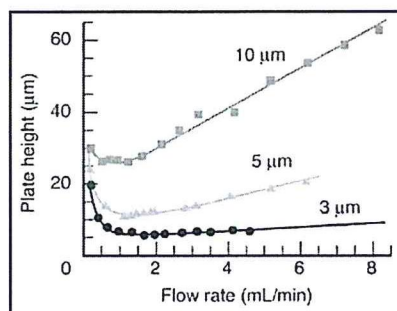
(ii) at pH 3.5?

(1)

(b) Figure (b) is also called a window diagram and is used to find the optimum separation by plotting α for each pair of solutes. Explain, using this figure, what would the optimum mobile phase pH be to obtain the best chromatographic separation of these 4 components.

(4)

3.3 The following figure shows the effect of flow rate on the column's plate height.



(a) In the Van Deemter Equation: $H = A + B/u + Cu$, which parameter is affected by changing the flow rate of a chromatographic system and what is that parameter called? (2)

(b) Explain the change (i.e. decreasing or increasing trend) observed in the size of the plate height when increasing the flow rate. (2)

(c) Which optimum flow rate would you use to obtain the best efficiency with the three particle sizes in the figure above and why? (2)

(d) Assuming you have two columns with particle sizes 3 μm and 10 μm . Which one would you choose to carry out your analysis (assuming all other characteristics are similar for both columns) and why? (3)

3.4 A 1.5 m column has 45000 plates per meter. Analyte A has a retention time of 16.3 minutes, analyte B has a retention time of 17.1 minutes and the dead (or void) time is 5.14 minutes.

(a) What is the resolution for A and B on this column? (4)

(b) What is the selectivity of the column? (2)

Question 4 [25]

4.1 Briefly explain how solutes separate in a mixture when using the following chromatographic techniques

(a) Adsorption chromatography (2)

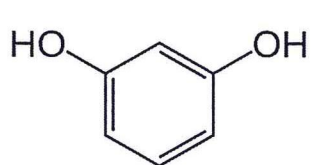
(b) Partition chromatography (2)

(c) Ion-exchange chromatography (2)

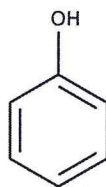
(d) Size-exclusion chromatography (2)

4.2 Name and briefly explain the different injection systems used in GC. (6)

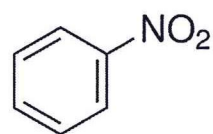
4.3 Two HPLC systems are defined below, and the following compounds are to be separated



Resorcinol



Phenol



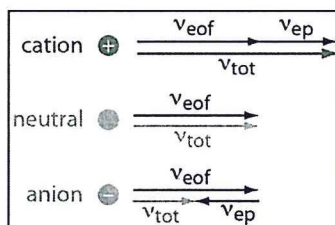
Nitrobenzene

HPLC system 1: uses silica stationary phase with hexane as the mobile phase.

HPLC system 2: uses C₁₈ stationary phase with a mobile phase consisting of 30 % water and 70% acetonitrile.

- Name the mode of separation (normal phase / reverse phase) using system 1. (1)
- Name the mode of separation (normal phase / reverse phase) using system 2. (1)
- Predict the elution order for the three compounds in a chromatogram using system 2 and explain your choice. (3)

4.4 Given the diagram below, explain the elution order of cations, neutral solutes and anions in electrophoresis.



(6)

END

Physical Constants

Gas constant	R	= 8.315 J K ⁻¹ mol ⁻¹ = 8.315 kPa dm ³ K ⁻¹ mol ⁻¹ = 8.315 Pa m ³ K ⁻¹ mol ⁻¹ = 8.206 x 10 ⁻² L atm K ⁻¹ mol ⁻¹
Boltzmann constant	k	= 1.381 x 10 ⁻²³ J K ⁻¹
Planck constant	h	= 6.626 x 10 ⁻³⁴ J s ⁻¹
Faraday constant	F	= 9.649 x 10 ⁴ C mol ⁻¹
Avogadro constant	L or N_A	= 6.022 x 10 ²³ mol ⁻¹
Speed of light in vacuum	c	= 2.998 x 10 ⁸ m s ⁻¹
Mole volume of an ideal gas	V_m	= 22.41 L mol ⁻¹ (at 1 atm and 273.15 K) = 22.71 L mol ⁻¹ (at 1 bar and 273.15 K)
Elementary charge	e	= 1.602 x 10 ⁻¹⁹ C
Rest mass of electron	m_e	= 9.109 x 10 ⁻³¹ kg
Rest mass of proton	m_p	= 1.673 x 10 ⁻²⁷ kg
Rest mass of neutron	m_n	= 1.675 x 10 ⁻²⁷ kg
Permittivity of vacuum	ϵ_0	= 8.854 x 10 ⁻¹² C ² J ⁻¹ m ⁻¹ (or F m ⁻¹)
Gravitational acceleration	g	= 9.807 m s ⁻²

Conversion Factors

1 W	= 1 J s ⁻¹
1 J	= 0.2390 cal = 1 N m = 1 V C = 1 Pa m ³ = 1 kg m ² s ⁻²
1 cal	= 4.184 J
1 eV	= 1.602 x 10 ⁻¹⁹ J
1 L atm	= 101.3 J
1 atm	= 1.013 x 10 ⁵ N m ⁻² = 1.013 x 10 ⁵ Pa = 760 mmHg
1 bar	= 1 x 10 ⁵ Pa
1 L	= 10 ⁻³ m ³ = 1 dm ³
1 Angstrom	= 1 x 10 ⁻¹⁰ m = 0.1 nm = 100 pm
1 micron (μ)	= 10 ⁻⁶ m = 1 μ m
1 Poise	= 0.1 Pa s = 0.1 N sm ⁻²
1 ppm	= 1 μ g g ⁻¹ = 1 mg kg ⁻¹ = 1 mg L ⁻¹ (dilute aqueous solutions only)

Selected Formulae

$$R_{AB} = \frac{t_{r,B} - t_{r,A}}{0.5(w_B + w_A)} = \frac{2\Delta t_r}{w_B + w_A} \quad R_{AB} = \frac{\sqrt{N}}{4} \times \frac{\alpha - 1}{\alpha} \times \frac{k_B}{1 + k_B}$$

$$k = \frac{1 - \frac{t_m}{t_r}}{\frac{t_m}{t_r}} = \frac{t_r - t_m}{t_m} = \frac{t_r'}{t_m}$$

$$\alpha = \frac{k_B}{k_A} = \frac{t_{r,B} - t_m}{t_{r,A} - t_m}$$

$$N = 16 \left(\frac{t_r}{w} \right)^2$$

$$q = nF$$

$$\Delta G = -nFE$$

$$I = E/R$$

$$E = E^0 - RT/nF \ln [B]^b/[A]^a$$

$$E \text{ (for ISE): } E_{\text{cell}} = K + 0.05916/z \log[A]$$

$$E = h\nu \text{ (or } E = hc/\lambda)$$

$$A = -\log T = \log P_0/P \quad \text{and} \quad A = \epsilon bc$$