



**NAMIBIA UNIVERSITY
OF SCIENCE AND TECHNOLOGY**

**FACULTY OF HEALTH, NATURAL RESOURCES AND APPLIED SCIENCES
SCHOOL OF NATURAL AND APPLIED SCIENCES**

DEPARTMENT OF BIOLOGY, CHEMISTRY AND PHYSICS

QUALIFICATION: BACHELOR OF SCIENCE	
QUALIFICATION CODE: 07BOSC	LEVEL: 7
COURSE CODE: MSC701S	COURSE NAME: MOLECULAR SPECTROSCOPY AND CHEMICAL SEPARATION METHODS
SESSION: JULY 2023	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

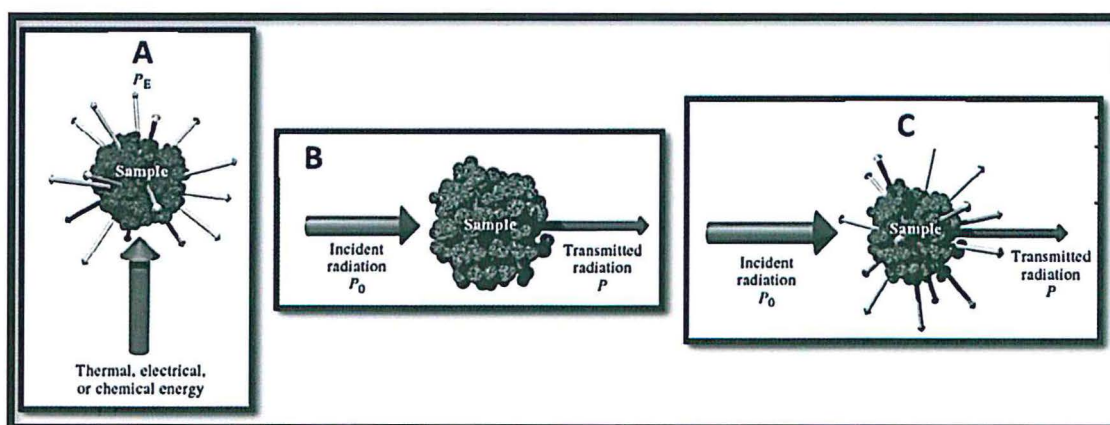
[25]

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)

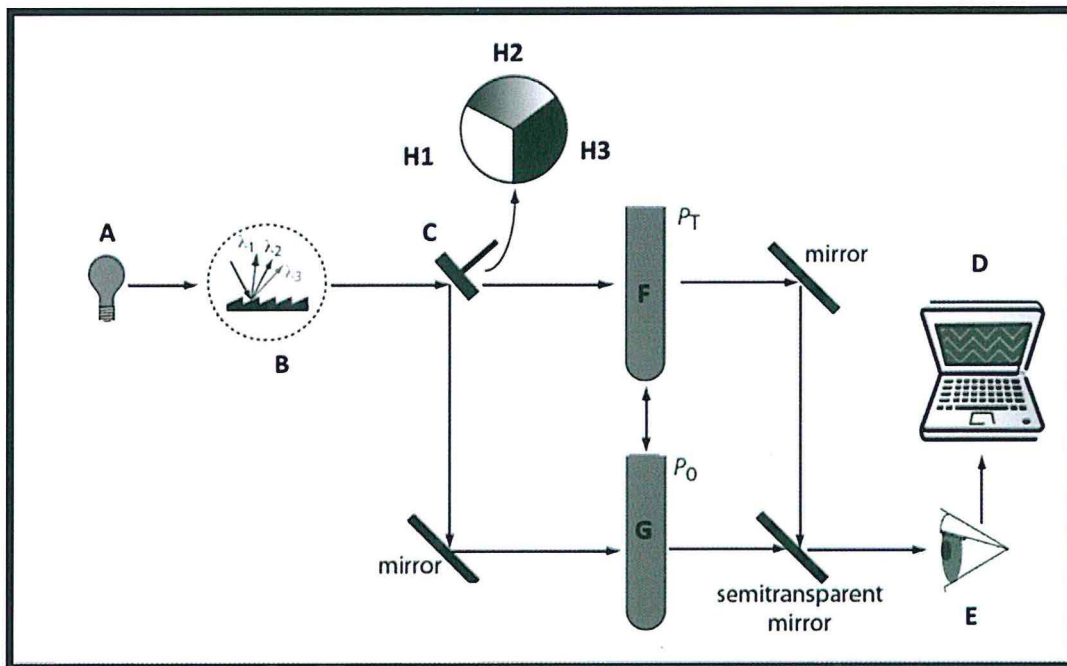
1.4 Differentiate between

- (a) Continuum and line sources of radiation (with an example for each). (4)
- (b) Monochromator and polychromator. (4)

Question 2

[25]

2.1 The following diagram represents a double-beam spectrophotometer. Some of the labels in the diagram have been hidden and replaced by letters A to H.



In this instrument, the device labelled "C" can be switched to the three positions shown in H1 to H3. Provide the name of that device and clearly explain its role and the purpose of these positions. (5)

2.2 The following table shows the temperatures obtained with different combination of fuels and oxidants in flame atomic absorption spectrometry (AAS).

Fuel	Oxidant	Temperature ($^{\circ}\text{C}$)
Natural gas	Air	1700 – 1900
Natural gas	Oxygen	2700 – 2800
Hydrogen	Air	2000 – 2100
Hydrogen	Oxygen	2550 – 2700
Acetylene	Air	2100 – 2400
Acetylene	Oxygen	3050 – 3150
Acetylene	Nitrous oxide	2600 - 2800

(a) Which combination of the fuels above will produce better excitation of atoms? Use the Boltzmann equation to support your choice. (5)

(b) What analytical parameter(s) is (are) improved with a better atomization efficiency? (2)

(c) Besides the flame, name three other means of atomization used in AAS. (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.

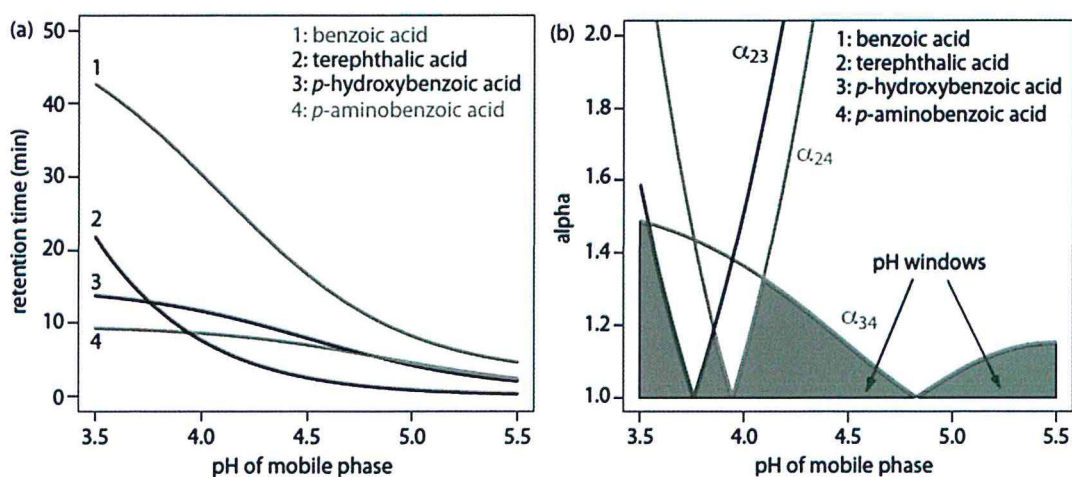
λ (nm)	Slit width (nm)	mg Cr/L giving $A = 0.20$	P_0 (relative)
357.9	0.2	2.5	40
425.4	0.2	12	85
429.0	0.5	20	100
520.5	0.2	1500	15
520.8	0.2	500	20

- (a) For the Cr hollow cathode lamp in the table above, which wavelength provides the best sensitivity? Explain your answer. (2)
- (b) 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)
- (c) The emission spectrum from a lamp includes, besides emission lines for the analyte, additional emission lines (from the metallic cathode impurities and the filler gas). What is the main inconvenience of these additional lines? (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 .



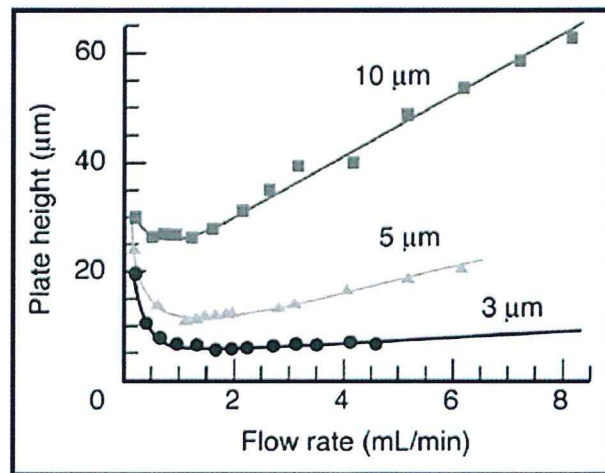
- (a) What unwanted situations would occur if the analysis was carried using a mobile phase

(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. Using this figure, explain what the optimum mobile phase pH would 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, $H = A + B/\mu + C\mu$, 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 (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

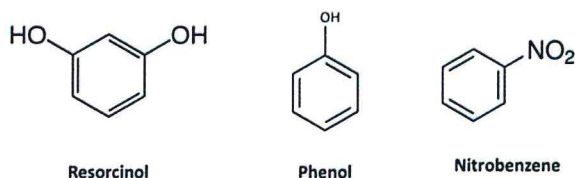
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4.1 Briefly explain how solutes separate in a mixture when using the following chromatographic techniques:

- (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

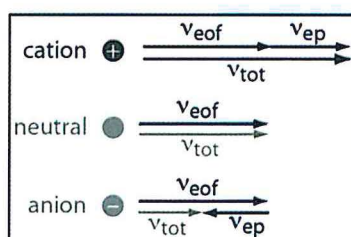


System 1: silica stationary phase with hexane as the mobile phase.

System 2: C₁₈ stationary phase with a mobile phase of 30 % water/70% acetonitrile.

- a) Name the mode of separation (normal phase / reverse phase) using system 1. (1)
- b) Name the mode of separation (normal phase / reverse phase) using system 2. (1)
- c) 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	$= 8.315 \text{ J K}^{-1} \text{ mol}^{-1}$ $= 8.315 \text{ kPa dm}^3 \text{ K}^{-1} \text{ mol}^{-1}$ $= 8.315 \text{ Pa m}^3 \text{ K}^{-1} \text{ mol}^{-1}$ $= 8.206 \times 10^{-2} \text{ L atm K}^{-1} \text{ mol}^{-1}$
Boltzmann constant	k	$= 1.381 \times 10^{-23} \text{ J K}^{-1}$
Planck constant	h	$= 6.626 \times 10^{-34} \text{ J s}^{-1}$
Faraday constant	F	$= 9.649 \times 10^4 \text{ C mol}^{-1}$
Avogadro constant	L or N_A	$= 6.022 \times 10^{23} \text{ mol}^{-1}$
Speed of light in vacuum	c	$= 2.998 \times 10^8 \text{ m s}^{-1}$
Mole volume of an ideal gas	V_m	$= 22.41 \text{ L mol}^{-1}$ (at 1 atm and 273.15 K) $= 22.71 \text{ L mol}^{-1}$ (at 1 bar and 273.15 K)
Elementary charge	e	$= 1.602 \times 10^{-19} \text{ C}$
Rest mass of electron	m_e	$= 9.109 \times 10^{-31} \text{ kg}$
Rest mass of proton	m_p	$= 1.673 \times 10^{-27} \text{ kg}$
Rest mass of neutron	m_n	$= 1.675 \times 10^{-27} \text{ kg}$
Permittivity of vacuum	ϵ_0	$= 8.854 \times 10^{-12} \text{ C}^2 \text{ J}^{-1} \text{ m}^{-1}$ (or F m^{-1})
Gravitational acceleration	g	$= 9.807 \text{ m s}^{-2}$

Conversion Factors

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

Selected Formulae

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

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

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

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

$$q = nF$$

$$\Delta G = -nFE$$

$$I = E/R$$

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

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

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

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