



NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY

FACULTY OF SCIENCE AND TECHNOLOGY EDUCATION

DEPARTMENT OF SCIENCE, MATHEMATICS AND TECHNOLOGY EDUCATION

ANALYTICAL CHEMISTRY (PST6144)

Main Examination Paper

NOVEMBER 2024

This Examination Paper consists of 5 pages

Time Allowed: 3 hours  
Total Marks: 100  
Special Requirements: Periodic Table, Calculator  
Internal Examiner: Mr SA Phiri  
External Examiner: Dr SJ Mpofu

**INSTRUCTIONS**

1. Answer **any five** questions. Each question carries **20 marks**.
2. Show all your steps clearly in any calculation.
3. Important data is at the back.

**MARK ALLOCATION**

QUESTION	MARKS
1	20
2	20
3	20
4	20
5	20
6	20
<b>TOTAL POSSIBLE MARKS</b>	<b>100</b>

1. a) Benzene and toluene were separated on a 12.5 cm long HPLC column. The retention times for benzene and toluene were 7.3 and 8.4 minutes respectively. The retention time for unretained peak is 2.1 min. the peak width were 0.74 for benzene and 0.87 min for toluene. The volume of the stationary phase is 1.37 ml and that of the mobile phase 1.43 ml.

$$V_s=1.37 \text{ ml}; V_m=1.43 \text{ ml}$$

$$t_{R\text{Benzene}}=7.3 \text{ min}; t_{R\text{Toluene}}=8.4 \text{ min}; t_m=2.1 \text{ min}$$

$$w_{\text{Benzene}}=0.74 \text{ min } w_{\text{Toluene}}=0.87 \text{ min}; L=12.5 \text{ cm}$$

- i. Calculate the phase ratio in the column. [2]
- ii. What are the capacity factors for both compounds? [4]
- iii. What is the distribution coefficient for both substances? [4]
- iv. Calculate the selectivity factor. [2]

b) Describe the following terms:

- i. Phase ratio
- ii. Retention time
- iii. Capacity factor
- iv. Selectivity factor. [8]

2. a) An HPLC analysis was conducted for caffeine on “Super-Extra-Energy Formula 2.2 with ‘Hyperdrive Now!’ sports drink. A 10.1 ppm methanol IS standard was introduced to both in the sample and a standardized solution of 304 ppm of caffeine. The results measured by a diode array detector at each  $\lambda_{\text{max}}$  for the absorption of methanol are summarized in the table. What is the concentration of caffeine in the sports drink?

	IS	Caffeine
Sample	23141	52777
304 ppm caffeine standard	28441	77313

[10]

- b) i. Describe two types of peak tailing comparing them with the Gauss peak [6]
- ii. State and describe 2 causes of peak asymmetry. [4]

3. a) A chromatographic separation of four compounds gave the following results:

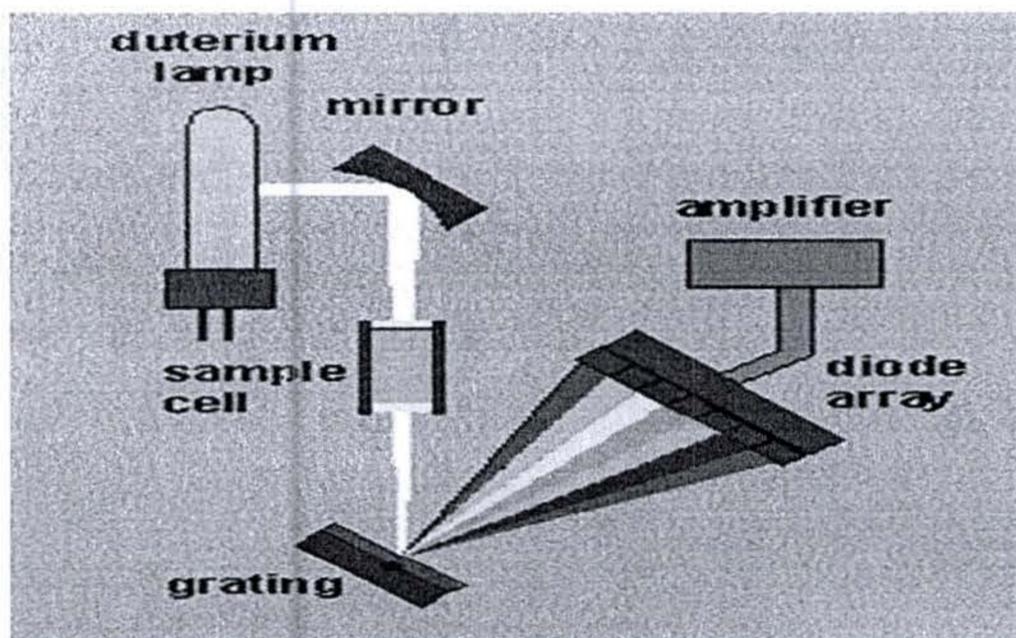
Analyte	1	2	2	4
Signal	0.072	0.046	0.061	0.041
Retention Time	120sec	255	310	608
Peak Width at Base	28sec	70	55	98

The dead time ( $t_m$ ) is 16.7sec.

- i. What is the capacity factor for analyte 4?
- ii. What are the number of plates for the column based on analyte 4?
- iii. What is the resolution for analyte 3 and 4? [15]

b) What is considered when purchasing or choosing an HPLC pump? [5]

4. a)



**Diagram of a Photodiode Array Detector (PDA)**

- i. Given the above diagram describe the working principle of the PDA.
- ii. A typical photodiode array has 512 diodes. Give reasons for this.
- iii. Name a typical light source in a PDA [10]

b) i. Describe the differences between gas-liquid chromatography and gas-solid chromatography. [5]

- ii. Discuss variables that lead to:

1. Band broadening
  2. Band separation in gas-liquid chromatography [5]
5. a) With the aid of diagrams, briefly describe:
- i. Affinity Chromatography
  - ii. Ion exchange Chromatography
  - iii. Thin layer Chromatography
  - iv. Paper Chromatography [12]
- b) What are the principal advantages and principal limitations of each of the detectors?
- i. Thermal conductivity
  - ii. Flame ionization [8]
6. a) i. Describe the differences between thin layer chromatography and paper chromatography. [6]
- ii. What is the aim of derivatization in GC? [4]
- b) Which ionization techniques would you prefer for the analysis of the following organic compounds.
- i. Anthracene
  - ii. A large protein
- Give reasons. [10]

END OF PAPER

1 H Hydrogen 1.008																	2 He Helium 4.003	
3 Li Lithium 6.94	4 Be Beryllium 9.012											5 B Boron 10.81	6 C Carbon 12.011	7 N Nitrogen 14.007	8 O Oxygen 15.999	9 F Fluorine 18.998	10 Ne Neon 20.180	
11 Na Sodium 22.990	12 Mg Magnesium 24.305											13 Al Aluminum 26.982	14 Si Silicon 28.085	15 P Phosphorus 30.974	16 S Sulfur 32.06	17 Cl Chlorine 35.45	18 Ar Argon 39.948	
19 K Potassium 39.098	20 Ca Calcium 40.078	21 Sc Scandium 44.956	22 Ti Titanium 47.867	23 V Vanadium 50.942	24 Cr Chromium 51.996	25 Mn Manganese 54.938	26 Fe Iron 55.845	27 Co Cobalt 58.933	28 Ni Nickel 58.693	29 Cu Copper 63.546	30 Zn Zinc 65.38	31 Ga Gallium 69.723	32 Ge Germanium 72.630	33 As Arsenic 74.922	34 Se Selenium 78.97	35 Br Bromine 79.904	36 Kr Krypton 83.798	
37 Rb Rubidium 85.468	38 Sr Strontium 87.62	39 Y Yttrium 88.906	40 Zr Zirconium 91.224	41 Nb Niobium 92.906	42 Mo Molybdenum 95.95	43 Tc Technetium [97]	44 Ru Ruthenium 101.07	45 Rh Rhodium 102.906	46 Pd Palladium 106.42	47 Ag Silver 107.868	48 Cd Cadmium 112.414	49 In Indium 114.818	50 Sn Tin 118.710	51 Sb Antimony 121.760	52 Te Tellurium 127.60	53 I Iodine 126.904	54 Xe Xenon 131.293	
55 Cs Cesium 132.905	56 Ba Barium 137.327	* 57 - 70	71 Lu Lutetium 174.967	72 Hf Hafnium 178.49	73 Ta Tantalum 180.948	74 W Tungsten 183.84	75 Re Rhenium 186.207	76 Os Osmium 190.23	78 Ir Iridium 192.217	79 Pt Platinum 195.084	80 Au Gold 196.997	81 Hg Mercury 200.592	82 Tl Thallium 204.38	83 Pb Lead 207.2	84 Bi Bismuth 208.980	85 Po Polonium [209]	86 At Astatine [210]	86 Rn Radon [222]
87 Fr Francium [223]	88 Ra Radium [226]	** 89 - 102	103 Lr Lawrencium [262]	104 Rf Rutherfordium [267]	105 Db Dubnium [270]	106 Sg Seaborgium [269]	107 Bh Bohrium [270]	108 Hs Hassium [270]	109 Mt Meitnerium [278]	110 Ds Darmstadtium [281]	111 Rg Roentgenium [281]	112 Cn Copernicium [285]	113 Nh Nihonium [286]	114 Fl Flerovium [289]	115 Mc Moscovium [289]	116 Lv Livermorium [293]	117 Ts Tennessine [293]	118 Og Oganesson [294]

5

\*Lanthanide series

57 La Lanthanum 138.905	58 Ce Cerium 140.116	59 Pr Praseodymium 140.908	60 Nd Neodymium 144.242	61 Pm Promethium [145]	62 Sm Samarium 150.36	63 Eu Europium 151.964	64 Gd Gadolinium 157.25	65 Tb Terbium 158.925	66 Dy Dysprosium 162.500	67 Ho Holmium 164.930	68 Er Erbium 167.259	69 Tm Thulium 168.934	70 Yb Ytterbium 173.045
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\*\*Actinide series

89 Ac Actinium [227]	90 Th Thorium 232.038	91 Pa Protactinium 231.036	92 U Uranium 238.029	93 Np Neptunium [237]	94 Pu Plutonium [244]	95 Am Americium [243]	96 Cm Curium [247]	97 Bk Berkelium [247]	98 Cf Californium [251]	99 Es Einsteinium [252]	100 Fm Fermium [257]	101 Md Mendelevium [258]	102 No Nobelium [259]
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