THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme / Stand-alone courses in Chemistry
Level 5 –Assignment Test 2 – 2015 / 2016

CMU 3128/CME 5128 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

Duration: One hour
Date and time: 12th November, 2016
          2.30 p.m. to 3.30 p.m.

Reg. No..

Question number	marks
1	
2	
Total	

Instructions to students

Answer all questions in the spaces given. Additional sheets will not be marked.

1. (i) Explain the term "Mass transfer" in column chromatography (meaning, at least one factor affecting and how it affects separation).

(14 marks)

(ii) Name the principle of separation and the stationary phase in paper chromatography.

(06 marks)

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(iii) (a) What is the reason for not considering Electrophoresis as a chromatographic method?

(b) List two characeristics of molecules that affects their rate of migration in Gel-Electrophoresis.

(12 marks)

(iv) The following data were obtained from HPLC using a mixture of methanol (75%) and water (25%) as the mobile phase which gave a retention time of 2.0 minutes. The column was packed with silica.

Compound	Retention time (minutes)
X	5.6
Y	6.4

(a) Calculate the selectivity factor for X and Y and comment on the separation of X and Y from each other.

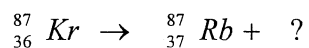(b) Which is more polar, X or Y? Give reasons for your answer.

(c) Suggest two ways to increase the efficiency of separation.

(24 marks)

2. (i) Briefly explain the term "dead time" of a detector.

(07 marks)

(ii) Complete the following nuclear equation.

$$^{87}_{36} Kr \rightarrow \ ^{87}_{37} Rb + \ ?$$

(03 marks)

(iii) State two properties of the radiation emitted in the reaction in (ii).

(04 marks)

(iv) (a) State one advantage of **Geiger-Muller counter.**

(b) State **two disadvantages of Geiger-Muller counter** and explain **how they are overcome** by the Gas flow counter.

(16 marks)

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(v)   A 20.0 mg of "Z" labelled with C^{14} with specific activity of 30 cpm/mg was added to a sample containing "Z" with some other organic compounds. After equilibration, a 40.0 mg of "Z" was separated from the mixture and it showed an activity of 200 cpm/mg. Calculate the **weight of Z** in the sample.

(14 marks)

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(01)

(i)   Mass transfer is the total resistance to the mass transfer in column chromatography.

$$C = C_S + C_M$$

C_S – resistance to the mass transfer between the stationary phase and mobile phase

C_M – resistance to the mass transfer within the mobile phase itself

Factors affecting the mass transfer:-

- Thickness of the stationary phase - Higher the thickness higher will be the resistance
- Diffusion coefficient – (either in mobile phase or the stationary phase)
  Higher the diffusion coefficient in the mobile phase or the stationary phase, lower will be the resistance
- Average diameter of particles- Higher the diameter, higher will be the resistance

Higher the mass transfer or the resistance, lower will be the efficiency of separation.

(ii)   In Paper Chromatography: Principle – partition; Stationary phase – water

(iii)   (a) There is no mobile phase.

  (b) Properties affecting the rate of travelling:
  - size and shape of molecules
  - The net charge of molecule

(iv)   (a) Selectivity Factor $(\alpha) = \dfrac{t_y - t_m}{t_x - t_m}$

$$= \frac{6.4 - 2}{5.6 - 2} \qquad = \frac{4.4}{3.6} = \mathbf{1.22}$$

Separation: high $(\alpha > 1.2)$.

 (b) X is less polar than Y.

    Reason: The retention time of X is less indicating that X had come out quickly with the mobile phase which is less polar than the stationary phase. Less polar compounds dissolve more in less polar solvents and carried away (or polar compounds dissolve more in more polar stationary phase resulting a higher retention time).

(c) Ways of increasing the efficiency of separation:-
- Make mobile phase less polar
- Change the rate of flow of the mobile phase
- Change the stationary phase (or use a column) with a more polar compound
- Use a column having less particle size
- Use a longer column

(02)

(i) Dead time is the time duration at which the radiation is not counted by the detector.

(ii) $^{87}_{36}Kr \longrightarrow {}^{87}_{37}Rb + {}^{0}_{-1}e$ (or β)
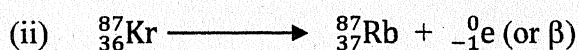
(iii) Properties of β rays: High penetration power; Light; Less initial energy but have more energy than α rays; therefore, more dangerous than α rays.

(iv) (a) Advantages: Simple; Effective

(b) Disadvantages:
- Detection is limited to high energy β, low energy δ and X rays.
- High energy γ particles pass through without interacting with the gas thus cannot be detected.
- Cannot distinguish between radiations of different energies.
- α rays cannot be detected.

The above disadvantages are overcome by placing the sample inside the gas chamber in Gas Flow Counter.

(v) **The question should be corrected as follows:**

"After equilibration, a 40.0 mg of "Z" was separated from the mixture and it showed an activity of **200 cpm (and not 200 cpm/mg)**.

$$m_x = \frac{R_t}{R_s} m_s - m_t \qquad R_t = 30 \frac{cpm}{mg} \times 20\ mg \qquad \mathbf{R_s = 200\ cpm}$$

$$\mathbf{m_x = \left(\frac{30 \times 20}{200} \times 40\right) - 20} \qquad m_t = 20\ mg \qquad m_s = 40\ mg$$

$$\mathbf{= 120 - 20 = 100\ mg}$$

* Due to the misprint in the question, marks were given only for the equation, R_t, m_t and m_s.

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