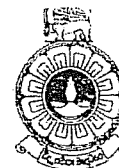


THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme / Stand-alone courses in Chemistry
Level 5 – Assignment Test 2 – 2016 / 2017



CMU 3128/CME 5128 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

Duration: One hour

Date and time: 5th November, 2017
 2.30 p.m. to 3.30 p.m.

Reg. No.

Question number	Marks
1	
2	
3	
Total	

Instructions to students

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

1. A mixture having the compounds A and B only was injected to a gas chromatograph (with a carrier gas) for separation. The dead time was 10.00 min. Results obtained are given in the following Table.

Parameter	Compound A	Compound B
Retention time (min.)	26.00	28.00
Peak area (arbitrary units)	321	565
Width of peak base (min.)	1.2	0.6

- (i) Do you think that the two peaks of A and B are well resolved? Justify your answer with suitable calculations.

(08 marks)

- (ii) If the gas chromatograph was run in reverse phase, what is more polar out of A and B? Give reasons for your answer.

(10 marks)

- (iii) State **two** important properties that compounds A and B should have in order to use gas chromatography for separation.

(06 marks)

- (iv) Calculate the percentage of A in the mixture.

(04 marks)

- (v) When the above separation was carried out having the same conditions but using a different detector, ratio of the peak areas obtained were different from the results shown in the above table. How do you overcome this problem?

(08 marks)

2. (i) What is meant by “Total capacity” of a strong cation exchanger?

(06 marks)

- (ii) A factory runoff was containing an equimolar concentration of ions X^+ and Y^{2+} . In order to separate the Y^{2+} from X^+ , a sample (10.0 mL) of effluent was passed through an open column packed with a strong cation exchanger (H^+ form) having a high total capacity. The effluent was titrated with 0.01 M NaOH and the end point obtained was 30.00 mL. Radius of the ions are given in the following Table.

Ion	Radius (°A)
X^+	1.74
Y^{2+}	1.74
Z^{2+}	1.89
E^+	1.25
D^{2+}	1.50

- (a) Which is eluted out of the two ions? Calculate the concentration of the eluted ion in the sample.

(08 marks)

- (b) Out of Z^{2+} , E^+ and D^{2+} , which will be suitable to elute the ion retaining in the column in the above separation? Justify your answer.

(10 marks)

- (iii) State **three** disadvantages of the physical structure of open columns used in liquid chromatography compared to HPLC.

(06 marks)

3. (i) What is the difference between Gamma Ray spectroscopy and other spectroscopic methods with respect to measurement?

(06 marks)

(ii) For what purpose the Isotope dilution method is used?

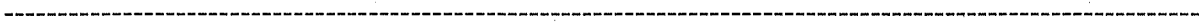
(06 marks)

(iii) Compare the mechanism behind Gas ionization detectors and scintillation detectors used in radioanalytical methods..

(12 marks)

(iv) What is meant by “Gradient elution” and “Isocratic elution”?

(10 marks)



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INSTRUMENTAL METHODS OF CHEMICAL ANALYSIS- CMU
3128- 2016/17

Continuous Assessment Test – II Answer guide

1. (i) Resolution (R) = $2 (|t_A - t_B|) / (W_A + W_B)$
 $= 2(|26 - 28|) / (1.2 + 0.6)$
 $= 2.2$

$R > 1.5$

Peaks of A and B are well resolved.

(ii) In reverse phase, the stationary phase is non polar and mobile phase

is

polar. Non polar substances dissolve more in non-polar solvents.

Therefore, more polar substances will dissolve more in the mobile phase and move fast resulting less retention time.

Retention time A < B. Hence, A is more polar than B.

(iii) Any two out of the following.

- Both should be volatile.
- Both should be resistant to temperature change (thermally stable).
- Should not react with both mobile and stationary phases.

(iv) %A = $[321 / (321+565)] \times 100\% = [321 / 886] \times 100\% = 36.2\%$

(v) Calculate the relative response factor for each component relative to a reference substance (either A/B) and get the corrected area value.

Corrected Area = Area (observed) / Relative response factor

2. (i) Total capacity = No. of equivalents that can be exchanged per unit weight of dry resin.

(ii) (a) X⁺ is eluted.

No. of moles of NaOH used = $(0.01/10^3) \times 30$

No. of moles of H⁺ exchanged = $(0.01/10^3) \times 30$

No. of moles of X⁺ exchanged with H⁺ = $(0.01/10^3) \times 30$

[X⁺] = $(0.01/10^3) \times 30 \times (10^3/$

10)

= 0.03M

(b) Z^{2+}

When ionic radius increases size of hydrated ion will decrease. The affinity depends on the effective charge.

Effective charge = charge / Size of hydrated ion

E^{2+} and D^{2+} - in both charge / Size of hydrated ion < that of Y^{2+}

- in Z^{2+} charge / Size of hydrated ion > that of Y^{2+}

(iii) Any 3 Advantages out of the following

- I. Particular size is large.
- II. Packed manually, therefore resolution is poor.
- III. Cannot be detected at the same time of running the Chromatogram.
- IV. The column may not have the optimum diameter.
- V. The column may not have the optimum column length.
- VI. Flow rate is adjusted manually thus may not be accurate.
- VII. Gradient elution is not possible.

3. (i) γ ray spectroscopy - Measure Energy directly; No need to calibrate.
Other spectroscopic methods - Measure light; Need calibration.

(ii) Isotope dilution method is used to determine the mass of a substance which cannot be purified or separated fully.

(iii) Gas ionization detectors - The inert gas absorbs radiation and produce ion pairs.

No. of ion pairs \propto No. of counts

Scintillation detectors - The scintillating material absorbs radiation and get excited. They relax and emit light of lower λ .

Amount of light emitted \propto No. of counts

(iv) Gradient elution - The composition of the mobile phase is changed with time while the chromatograph is running.

Isocratic elution - The composition of the mobile phase is not changed which the chromatograph is running. It is the same mobile phase used throughout.