King Saud University Department of Chemistry



Chemical Separation & Chromatographic Methods

- Q₁) Define [5 marks]
- (a) Chromatography
- (b) Stationary phase
- (c) Gradient elution
- (d) Reversed phase chromatography
- (e) Preconcentration

Q₂) Fill blanks [10 marks]

- (a) Extraction is a family of separation methods based on
- (b) A chromatogram is useful for both and quantitative analysis. The of the peak may serve to identify the components of the sample; while the heights or under the peaks provide a quantitative measurement of each component
- (c) To achieve a separation there must be at least one significant difference between the or properties of the analyte and interferent
- (d) The time it takes after sample injection for the analyte peak to reach the detector is called
- (e) We need separation methods for or
- (f) Separation technique based on mass (an example)
- (g) Based on the purpose of chromatography experiment, chromatographic methods can be categorized to and
- (h) Masking is a pseudo-separation method based on
- (i) Sublimation is a separation technique based on
- (j) In chromatography, mobile phase could be, liquid or a supercritical fluid, while stationary phase could be liquid or
- (k) In liquid-solid or adsorption chromatography the stationary phase is, while in gas-liquid chromatography the mobile phase is
- (I) Separation technique based on change in physical state (an example)
- (m) HPLC is an abbreviation for

Q₃) Circle the one correct answer from the choices listed [6 marks]

The simplest property that can be exploited in a separation is

- (a) mass
- (b) size
- (c) charge
- (d) partitioning

A student runs a sample of n-propanol through a gas chromatogram at 95°C. The n-propanol produces a peak after 4.2 minutes. The student then injects a mixture of unknown organic substances into the same column at the same temperature. There are peaks after 3.1, 4.2 and 7.4 minutes. From this information, it can be concluded that

- (a) the mixture has three components, one of which must be n-propanol
- (b) the mixture has at least three components, one of which must be n-propanol
- (c) the mixture has three components, but n-propanol is not one of them
- (d) the mixture has at least three components, one of which might be n-propanol

Of the following compounds, which would you expect to elute *first* from a gas chromatography column?

- (a) methanol
- (b) ethanol
- (c) n-propanol
- (d) n-butanol

Of the following compounds, which would you expect to elute *last* from a reverse-phase liquid chromatography column?

- (a) methanol
- (b) ethanol
- (c) n-propanol
- (d) n-butanol

In normal phase chromatography, there is a

- (a) the mobile phase is polar and the stationary phase is nonpolar
- (b) the mobile phase is nonpolar and the stationary phase is polar
- (c) both the mobile and the stationary phase are non-polar
- (d) both the mobile and the stationary phase are polar

Liquid chromatography methods include

- (a) liquid/liquid (partition) chromatography
- (b) liquid/solid (adsorption) chromatography
- (c) ion exchange and size exclusion chromatography
- (d) all of the above

Q₄) Describe chromatographic technique (briefly), and give an example and sketch if possible [3 marks]

Q₅) Describe the five major **GC** components and their functions, a diagram can be useful [5 marks]

Q₆) Modern chromatographic methods have many applications; list three of them [3 marks]

Q₇) List five variables affect method efficiency in chromatography [5 marks]

Q8) What are the main differences between

- (a) HPLC and GC [2 marks]
- (b) Separation and purification [2 marks]

 Q_9) A chromatograph of a two component mixture is shown below. The LC column is 250 mm long. The flow rate was 0.50 mL/min. Using the chromatograph to calculate the following, knowing that width of the peaks at half (w_{1/2}) for Glucose, Maltose and Maltotriose are 0.33, 0.67 and 1.32min, respectively



Calculate

- (a) The time of **Glucose** spends on the stationary phase [1 marks]
- (b) The retention factor for Maltotriose [1 marks]
- (c) The resolution between Maltose and Maltotriose [1 mark]
- (d) The selectivity factor for Maltose and Maltotriose [1 mark]
- (e) An average number of plates for the three components [3 mark]
- (f) An average plate height for the column [1 mark]
- (g) The standard deviation for Maltose [1 mark]

Related equations:

$$k' = \frac{t_R - t_M}{t_M}$$

$$\alpha = \frac{t_{R2} - t_M}{t_{R1} - t_M}$$

$$R_s = 2 \frac{t_{R2} - t_{R1}}{w_1 + w_2} = 1.18 \frac{t_{R2} - t_{R1}}{w_{(1/2)1} + w_{(1/2)2}}$$

$$H = \frac{L}{N}$$

$$N = (\frac{t_R}{\sigma})^2 = 16 (\frac{t_R}{w})^2 = 5.54 (\frac{t_R}{w_{(1/2)}})^2$$

Good Luck !! Course instructor Dr. Ahmad Aqel