Chapter 28

28-1

List the types of substances to which each of the following chromatographic methods is most applicable:

a) Gas-liquid

b) Liquid adsorption Ans: Nonpolar low to moderate molecular mass organics and particularly isomeric organic species (非極性低至中等分子量有機物，特別是異構有機物種類)

c) Liquid-liquid partition Ans: Molecular species that are nonvolatile or thermally unstable. (非揮發性或熱不穩定的分子種類)

d) Reverse-phase partition Ans: Most low to moderate molecular mass organic compounds that are nonvolatile or thermally unstable. (大多數低至中等分子量的有機化合物，非揮發性或熱不穩定)

e) Ion exchange

e) Gel permeation

)

e) Gas-solid

體)

e) . (高分子量

親水化合物)

https://microbenotes.com/gel-filtration-chromatography/

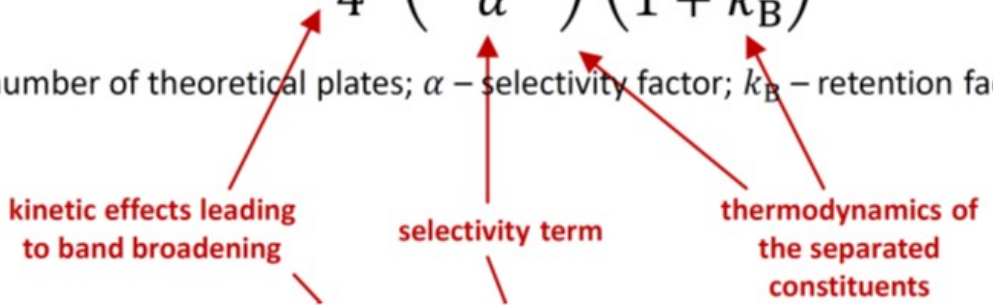
28-2

Describe three general methods for improving resolution in partition chromatography

Resolution:

$$R_s = \frac{\sqrt{N}}{4}\left(\frac{\alpha - 1}{\alpha}\right)\left(\frac{k_B}{1 + k_B}\right)$$

where: N – number of theoretical plates; α – selectivity factor; k_B – retention factor.



1. Adjustment of k_A and k_B by employing a multicomponent mobile phase and varying the ratio of the solvents to find an optimal mixture. (通過使用多組分流動相並改變溶劑的比例以找到最佳混合物來調節 k_A 和 k_B)
2. Variation in the chemical composition of the solvent system in such a way as to make α larger. (溶劑體系的化學組成的變化，使 α 變大)
3. Employing a different packing in which α is greater. (採用 α 更大的不同填料)

28-3

Describe a way to manipulate the retention factor of a solute in partition chromatography.

- Retention factor is used to compare migration rates of solutes in columns:

$$k_A = \frac{K_A V_S}{V_M}$$

or

$$k_A = \frac{(t_R)_A - t_M}{t_M}$$

where: K_A – distribution constant; V_S and V_M – volumes of the two phases; t_R – retention time; t_M – dead/void time.

In partition chromatography, k is conveniently varied by using a two (or more) component solvent system and varying the ratio of the solvents (描述一種在分配色譜中操縱溶質保留因子的方法。使用兩種（或更多種）組分溶劑系統並改變溶劑的比例，可方便地改變 k)

Question 28-4

How can the selectivity factor be manipulated in (a) GC and (b) LC?

$$\alpha = \frac{(t_R)_B - t_M}{(t_R)_A - t_M}$$

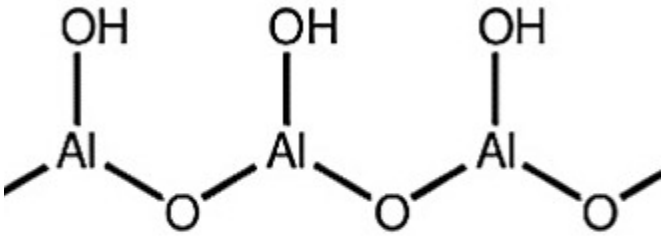
(a) In gas chromatography, α is generally varied by varying the column packing

(b) In LC, both column packing and chemical composition of the mobile phase can be varied to yield better α values.

Question 28-5

In preparing a hexane-acetone gradient for an alumina HPLC column, is it desirable to increase or decrease the proportion of hexane as the column is eluted?

Stationary phase: alumina packing polar
Hexane nonpolar
Acetone polar



increase the polarity of the mobile phase as the elution proceeds
proportion of hexane should decrease

沖提能力應由小至大，也就是說動相與固相之差異由大到小。

Question 28-6

What is meant by the linear-response range of a detector?

range of analyte concentration or mass over which the detector responds linearly

linear-response range = dynamic range

Question 28-7

Define:

- **isocratic elution**

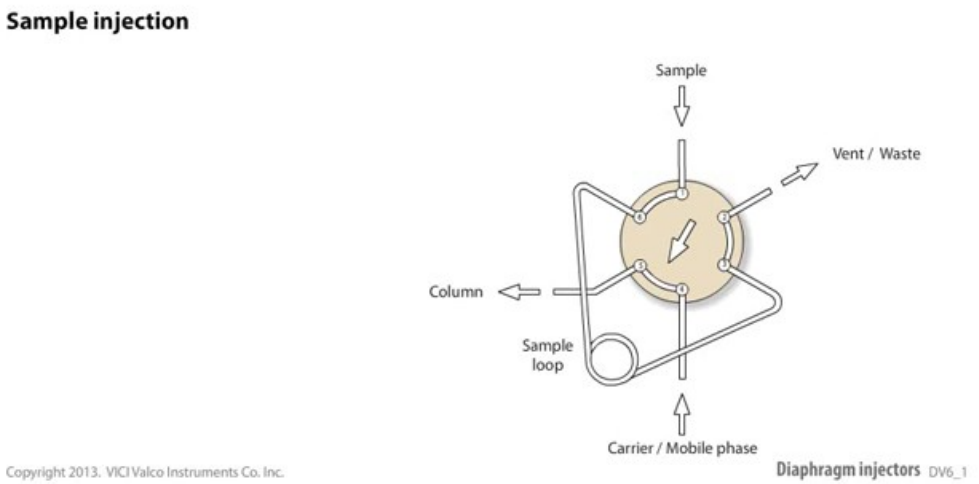
the solvent composition is held constant throughout the elution

- **gradient elution**

two or more solvents are used and the composition of the mobile phase is changed continuously or in steps as the separation proceeds

- **stop-flow injection**

the flow of solvent is stopped, a fitting at the head of the column is removed, and the sample is injected directly onto the head of the column. The fitting is then replaced and pumping is resumed.



https://www.youtube.com/watch?v=8-D-mMfDw7g

Question 28-7

Define:

- **reversed-phase packing**

a nonpolar packing that is used in partition chromatography with a relatively polar mobile phase

- **normal-phase packing**

the stationary phase is polar and the mobile phase is relatively nonpolar

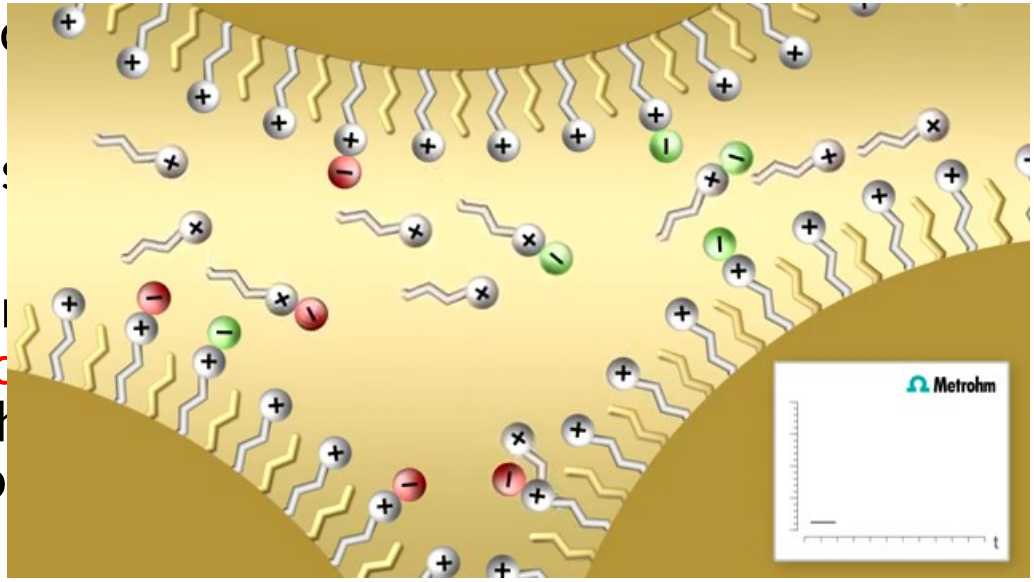
- **ion-pair chromatography**

注意與 Ion-exclusion chromatogra
的差異

Ion-pair chromatography allo
reversed-phase columns.

a large organic counter-ion is
pairing reagent.

separation is achieved either
ion-pair or as a result of elec
in solution and charges on th
adsorption of the organic co



Question 28-9

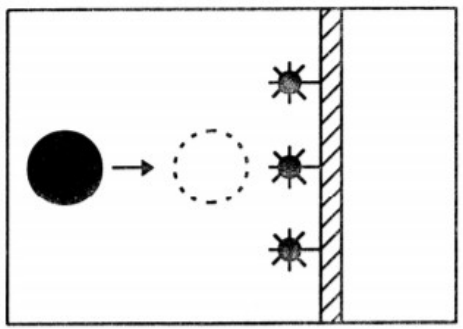
In what way are normal-phase partition chromatography and adsorption chromatography similar?

Adsorption chromatography(吸附層析法)

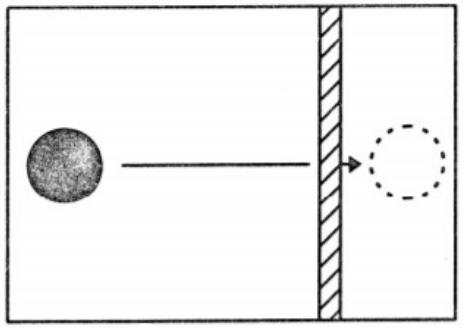
利用樣品在吸附劑（固定相）上的吸附能力強弱不同而得以分離的方法，稱為吸附層析法。

Partition chromatography(分配層析法)

利用樣品在惰性固體上的液體（固定相）中溶解度不同（即在二不互溶液體間之分配係數的差異），以萃取法而達到分離稱為分配層析法。



吸附層析法



分配層析法

類　型	流動相極性	固定液極性
正相分配層析法	非極性	極性
逆相分配層析法	極性	非極性

Question 28-12

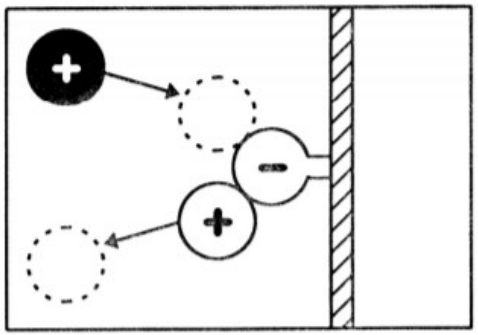
Describe the fundamental difference between adsorption and partition chromatography.

Question 28-13

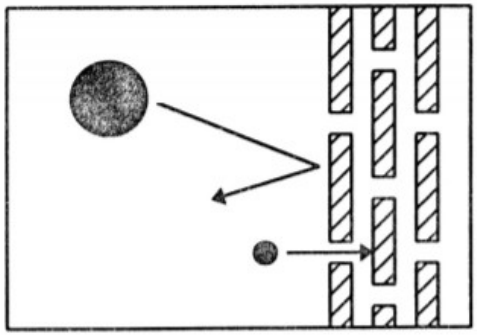
Describe the fundamental difference between ion-exchange and size-exclusion chromatography.

Ion-exchange chromatography (離子交換層析法):
利用試料成分在離子交換樹酯 (固定相) 表面上之親和力的差異來進行分離的層析法。

Size-exclusion chromatography (大小排除層析法):
利用分子大小不同來分離，又稱分子篩層析法、凝膠滲透層析法。



離子交換層析法



大小排除層析法

Question 28-14

What types of species can be separated by HPLC but not by GC?

Question 28-17

Mass spectrometry is an extremely versatile detection system for GC. However, interfacing an HPLC system to a mass spectrometer is a much more difficult task. Describe the major reasons why it is more difficult to combine HPLC with mass spectrometry than it is to combine GC with mass spectrometry.