Faculty of Pharmacy
Department of Pharmaceutical Chemistry and Pharmacognosy
Instrumental Analysis
Final Exam 08-05-2017   *Time 110 minutes*

Name

I)- Choose the best correct answer: (27 Marks)

1- Ethylenic $C=C$ has lower stretching frequency than $C=O$ due to ?
a- atomic masses   b- bond strength   c- a and b   d- None

2- Which of the following statements regarding column efficiency in HPLC is TRUE?
a- Efficiency decreased using longer columns; however, the analysis time usually increases
b- Efficiency increased using smaller particles, however the pressure usually increases.
c- Efficiency increased using longer columns; and the analysis time usually decreases
d- Efficiency increased using larger particles, however the pressure usually increases.

3-In NP-HPLC separation of barbiturates (barbital is more polar than Phenobarbital).
How the following parameters will change the retention factor of Barbital?
i. Decreasing the polarity of the stationary phase
a- increase   b- decrease   c- no change

ii. Decreasing the polarity of the mobile phase:
a- increase   b- decrease   c- no change

iii. Barbital will have lower t_R than phenobarbiatl: a- True   b-False

4- Which of the following detectors is not commonly used for HPLC?
a- Thermal conductivity   b- Mass spectrometry   c- Fluorescence
d- UV/Vis   e- Refractive index

5- Which of the following statements concerning SEC is incorrect?
a- Used in separation of proteins from peptides and molecular-weight determination
b- In gel permeation chromatography organic mobile phase is used
c- Larger molecules elute first
d- Retention is based on size/shape of the analyte and the pore size of the support

6- Which of the following is a type of planar chromatography?
a- GC   b- AC   c- IEC   d- SEC   e- TLC

7- Which of the following chromatographic techniques is most suitable for small,
nonvolatile, water-insoluble solutes?
a- GC   b- reverse phase (RPLC)   c- NPLC   d- SEC

8- Solutes present at high concentrations in samples subjected to chromatography will
exhibit
a- short retention times   b- large peak areas
c- small peak areas   d- long retention times

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9- Which of the following is incorrect regarding CC?

a- Selectivity coefficient is used to describe how well two solutes are separated.

b- The later eluting bands will spread more

c- Longitudinal diffusion is diffusion takes place along the axis of the column and contribu
peak broadening.

d-All are correct

10- Which of the following is incorrect regarding GC?

a- WCOT is less efficient than SCOT and both are more efficient than packed column.

b- Could be used to determine solvent residues in drug substances.

c- Temperature programming is better than isothermal separation for samples with wide boi
point range

d- The order of elution is related to boiling point where substances of high volatility elute fi

11- Which of the following is incorrect regarding HPLC?

a- Degassing of mobile phase could be carried out by sparging with He

b- ODS is one of the most common reversed phase used

c- Stationary phase type, Eluent additives, flow rate are parameters affecting selectivity

d- Increasing the capacity factor (up to10) improves separation

12- Which of the following statements regarding NMR spectroscopy is wrong?

a- The frequency at which a particular proton absorbs is determined by its elect
environment.

b- δ are larger when the frequencies of the radiation which induces the nuclear transitions ar
higher.

c- Chemical shifts are larger when shielding effects are greater.

d- All these atoms are active in NMR ^{14}N, ^{19}F and ^{2}H.

13- How many signals does the unsaturated ketone $(CH_3)_2CHCH_2C(O)CH=CH_2$ have
NMR and ^{13}C NMR spectra?

a- five ^1H signals and six ^{13}C signals

c- six ^1H signals and seven ^{13}C signals

b- six ^1H signals and six ^{13}C signals

d- five ^1H signals and seven ^{13}C signal

14- In gas chromatography, if two solutes with short retention times co-elute (i.e. an
resolved), what is the simplest way to attempt to resolve the peaks?

a- Use a different carrier gas

c- Use a lower column temperature

b- Use a higher column temperature

d- Use a more polar solvent

15-Using silica TLC plate for the separation of (1-RCl, 2-RCO$_2$CH$_3$, 3-ROH) which
following is correct regarding R$_f$ values

a- 1 < 2 < 3   b- 2 < 3 < 1

c- 3 < 2 < 1   d- 3 < 1 < 2

16- The IR- spectrum of Propyne is characterized by the following bands (cm^{-1})

a- 3300, 2985, 2150, 1450

c- 3300, 3100, 2980, 1400

b- 3500, 2980, 2250, 1450

d- 2920, 2180, 1425, 1375

17- In chromatography, the capacity factor is related to:

a- column length
c- equilibrium constant between the mobile and stationary phase

b- flow rate
d- all of the above

II- Give the best match for the terms in the first list with the characteristics in the second: (8 marks)

| A		B
1 ECD		It is used for separation of charged molecules.
2 Area under the peak		He
3 Eddy diffusion		Chromatogram parameter used in quantitative analysis
4 FID		charged molecules in solution migrate in response to an electrical field.
5 Electrochemical detector		Halogen containing pesticides
6 Adjusted retention time		Has a β-emitter causes ionization of the carrier gas
7 TCD		Monitor any compound that can undergo an oxidation or reduction
8 Electrophoresis		A process that leads to peak broadening due to the presence of multiple flow paths through column.
9 IEC		

III)- Complete the following statements (9 marks)

1- Example on nonpolar SP in GC is..
2- ....a graph showing the detectors response as a function of elution time
3-In AAS the .... is used as radiation source
4-In chemically bonded C_4 stationary phase, the strong mobile phase is ....
5- Non-specific elution in AC could be attained by...
6-A weak acid, HA, has a pKa of 6.5. IEC was used for separation using MP at pH 8.0. The SP would be...(type)
7- Near UV radiation wavelength range is.............................nm
8- In HPLC, ... is the common pump and .... is used for sample loading.

IV)- Answer the following questions

1- ^{1}H NMR spectrum of a compound whose molecular formula is $C_4H_{10}O$ showed peaks at 3.375 (quartet) and 1.150 (triplet). Deduce the structure of the compound. (2 marks)

2- Mention the name of the instrument and identify the main parts shown on its figure. (3 marks)

A.............................
B.............................
C.............................
D.............................
Name.............................

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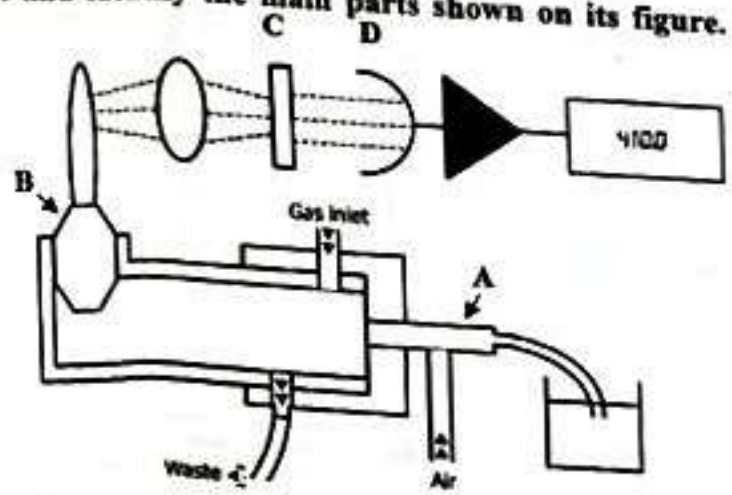
3) **Given the following chromatogram and a column length of 25 cm, flow rate 0.5 mL/min** ($W_{b\ for\ A}$ = 1.5 min; $W_{b\ for\ B}$ = 2 min):(7 marks)
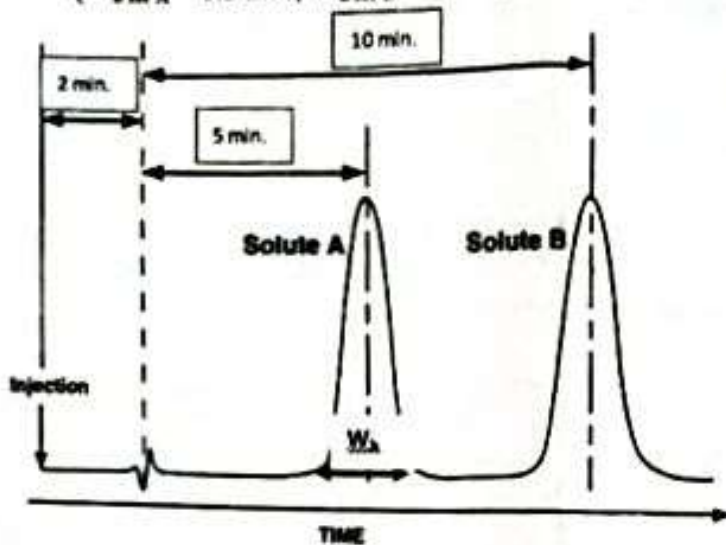
Calculate:
1-capacity factor for solutes B
2-number of theoretical plates for solute
3- plate height for solutes A
4- separation factor
5-how would you classify the performa of the column based on the resolution?
6- void volume

$$\alpha = \frac{t'_{r2}}{t'_{r1}} = \frac{k'_2}{k'_1} = \frac{K_2}{K_1} \qquad H = \frac{L}{N}$$

$$N = \frac{16t_r^2}{w^2} = \frac{5.55t_r^2}{w^2_{1/2}} \qquad k' = t'_r/t_0$$

$$\text{Resolution} = \frac{\Delta t_r}{w_{av}}$$

4- **Mention: a) the main steps in CC (2 marks). b) Factors affecting migration rate in electrophoresis (2 marks)**

5- **Draw diagram showing main units in GC instrument.(4 marks)**

6- **Indentify the molecule ($C_9H_{10}O_2$) whose spectra are provided with justification (6 marks)**