
	<b>TANTA UNIVERSITY</b> <b>FACULTY OF PHARMACY</b> <b>DEPARTMENT OF PHARMACOGNOSY</b>			
	<b>FINAL EXAM FOR THIRD YEAR CLINICAL STUDENTS</b>			
<b>COURSE TITLE:</b>	<b>QUALITY CONTROL OF HERBAL MEDICIN</b>		<b>COURSE CODE:</b> <b>PG606</b>	
<b>DATE:</b>	<b>25/5/2019</b>	<b>TERM:</b> <b>SECOND</b>	<b>TOTAL ASSESSMENT MARKS: 50</b>	<b>TIME ALLOWED: 2 HOURS</b>

**Part I****Chromatography .....(30 marks, 72 minutes)**

A- Choose only one correct answer and record your choice in the corresponding place in the answer sheet provided below: .....(12 Marks)

## Answer sheet

No.	a	b	c	d	e	No.	a	b	c	d	e	No.	a	b	c	d	e	
1						9						17						
2						10						18						
3						11						19						
4						12						20						
5						13						21						
6						14						22						
7						15						23						
8						16						24						

- Regarding to sephadex LH-20 all are true except;
  - More dense and has smaller pore size
  - More lipophilic and can separate non polar compounds
  - Adsorption forces play a greater role
  - It can separate water soluble compounds
  - It is isopropyl derivative of polyglucose.
- All Sugars can be separated by
  - Ion exchange chromatography after the formation of borate complex
  - Gas chromatography after silylation
  - Gel filtration
  - HPLC using any type of detectors
  - Both (a) and (b)
- To concentrate 30 ml of 10% protein to 20% solution by addition of
  - 5 gm. Sephadex LH-20
  - 6gm.of sephadex G25
  - 2gm. G50
  - All of the above
  - Both (b) and (c)
- Mixture of 30%  $AlCl_3$ , 30%NaCl and 40% $CaCl_2$  is separated by
  - Cations exchange resin and NaCl is eluted first
  - Cations exchange resin and  $CaCl_2$  is eluted later
  - Cations exchange resin and  $AlCl_3$  is eluted later
  - Both (a) and (b)
  - Both (a) and (c)

- 5- Neutralization of alkaline solution can be carried out by the use of  
 a-  $\text{Na}^+$  form cationic resin                      b- Any form of cationic resin  
 c-  $\text{Cl}^-$  form resin                                      d-  $\text{OH}^-$  form anionic resin  
 e- Both (b) and (c)
- 6- Regarding to the disruption coefficient all of the following are true except  
 a- Is increased in G.C. by increasing the temperature of the column  
 b- Is changed with increasing the rate of diffusion  
 c- It's change lead to broadening and asymmetry of peak  
 d- Is changed when we use high solute concentration  
 e- Is increased in gel filtration with aromatic and heterocyclic compounds
- 7- Factors affecting column performance all are true except;  
 a- Particle size not more than  $50 \mu$   
 b- High solute diffusivity in carrier gas  $\rightarrow$  broad peak  
 c- The velocity of the carrier gas  
 d- The efficiency is  $\uparrow$  by  $\downarrow$  the Column diameters  
 e- Increase the column temperature decrease the partition coefficient
- 8- Regarding to high solute diffusivity in carrier gas;  
 a- Gives broad peak                                      b- Is increased by increasing temperature  
 c- Is decreased by using more viscous mobile phase  
 d- Is increased by using He                              e- All of the above
- 9- The ability of a solute molecules to penetrate the gel matrix depends on;  
 a- Arrangement of atoms in space                      b- Molecular weight  
 c- The presence of aromatic or heterocyclic compounds  
 d- Whether branched or straight-chained                      e- Both (c) and (d)
- 10- For separation of glycerol from KCl we use;  
 a- Amphoteric resin and glycerol is eluted first  
 b- Cationic resin  $\text{K}^+$  form and KCl is eluted later  
 c- Donnan equilibrium and KCl is eluted first  
 d- Both (a) and (c)                                      e- All of the above
- 11- Regarding to multifunction strong cationic resins all are true except;  
 a- Can be prepared by Polycondensation of phenol with formaldehyde with the introduction of sulfonic acid group  
 b- Is unstable                                      c- Not affected by the change of the pH  
 d- Copolymerization of methyl acrylic acid with vinyl pyridine  
 e- Is considered as high capacity resin
- 12- Regarding to electron capture detectors all of the following are true except;  
 a- Nickel is present as a source of highly energetic electrons  
 b- Nickel is present to collect the emitted electrons from  $\beta$  rays  
 c- Decrease in signal rather than increase is measured  
 d- Is sensitive to halogenated compounds                      e- Is highly sensitive

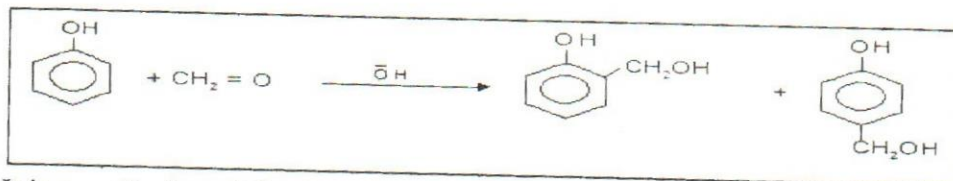
- 13- When we use gradient elution in HPLC we should use degasser with
- a- Any type of detectors
  - b- Refractive index detector only
  - c- TCD
  - d- Any type of detector except TCD
  - e- None of the above

- 14- Regarding to Gelay columns
- a- Should be open columns.
  - b- It is used for partition and adsorption chromatography
  - c- It is used for partition chromatography only
  - d- The liquid stationary phase is supported by inert support
  - e- Both (a) and (c)

- 15- The buffer is important in chromatographic separation because
- a- It promote the stability of compounds in gel filtration
  - b- It increases the difference in charge size in ion exchange chromatography.
  - c- It increases the volatility of compounds in GC.
  - d- Both (a) and (b)
  - e- All of the above

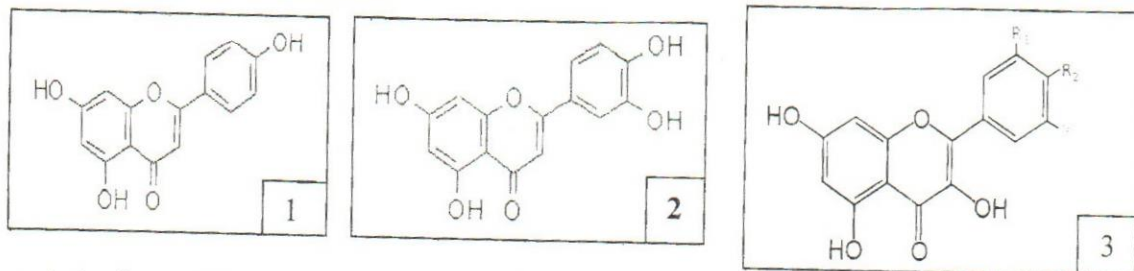
- 16- The adjustment of pH can be carried out by
- a- The used of buffer in case of gel filtration
  - b- NaCl. and hydrogen form resin to get acidic pH
  - c- NaCl. and anionic form resin to get alkaline pH
  - d- Both (a) and (b)
  - e- All of the above

- 17- Regarding to this reaction



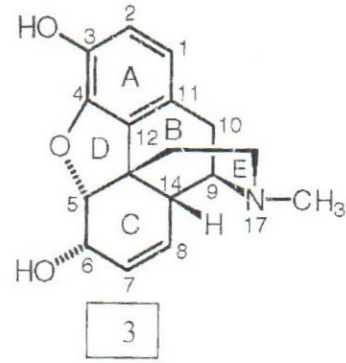
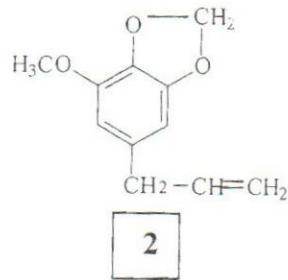
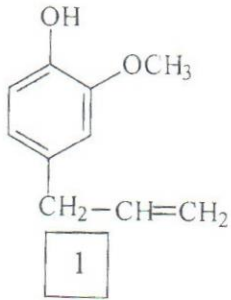
- a- It is a method used for the preparation of unbranched poly phenols
- b- The capacity of this polymer depends on the pH range
- c- Multifunction cationic resin can be prepared from this polymer
- d- All of the above
- e- Both (b) and (c)

- 18- This mixture can be separated by



- a- Anionic exchange resin and comp. 3 is eluted first
- b- Anionic exchange resin and comp. 2 is eluted later
- c- Reversed phase HPLC and comp. 2 is eluted first
- d- Both (b) and (c)
- e- All of the above

19- This mixture is separated by



- a- Anionic resin and comp.2 is eluted first  
 b- Anionic resin and comp.3 is eluted later  
 c- Cationic resin and comp.3 is eluted later  
 d- Both (a) and (b) e- All of the above

20- The affinity of substances to ions exchange chromatography is increased by All of the following except

- a- Increase the molecular weight to certain limit  
 b- Increase the charge size which suppress the concentration effect  
 c- Increase the adsorptivity of the compound  
 d- The presence of aromatic rings in the compounds  
 e- Increasing the valence

21-Regarding to void volume all of the following are true except

- a- Is equal to  $6 \text{ ml}$  if we use  $3 \text{ gm.}$  of sephadex LH-20  
 b- Distribution coefficient of the compounds present in it is equal to zero  
 c- Equal to  $V_t - (V_i + V_g)$   
 d- Contain the large particle size compounds  
 e- Dextran blue 2000 is used for the determination of it

22- The removal of salts from proteins can occur by using;

- a- Gel filtration where the protein is eluted first  
 b- Ampheteric Resins where protein is eluted first  
 c- Donnan equilibrium ion exchange resin where salt is eluted first  
 d- HPLC e- All of the above

23- Introduction of  $\text{CF}_3$  group to the compound cause

- a- Increase the thermal stability of the compounds  
 b- Improve the separation and reduce tailing  
 c- Increase the detector response especially ECD.  
 d- Increase the detector response especially FID.  
 e- All of the above

24- The capacity of the resin depends on

- a- Type of bound ions on the resin  
 b- The sample concentration and its ionic strength  
 c- Concentration of the ions to be exchanged  
 d- pH for all type of resin e- Both (a) and (c)

B- Write the letter (T) to the correct answer, and the letter (F) to the false one  
Correct the false one and write the correct answer in the specific place ..(8 marks)

NO.	Statements	Correct words	Letter
1	LH20 is considered as reversed phase chromatography		
2	Pyrolysis GC is used for the identification of highly sensitive polymers		
3	Multifunction strong cationic resins is more strong and stable than the unfunction one		
4	Sepharose gel is suitable for separation of very high molecular weight compounds		
5	open tubular columns it is used for partition chromatography only		
6	mass transfer cause radial transport of solute in opposite direction in both phases		
7	Detector used to detect all types of organic and inorganic compounds is ECD		
8	NPD, TCD and ECD are used for detection of halogenated compounds		
9	Thermoionic alkali bead detectors are not affected by the flame temperature		
10	$\beta$ ray emit thermal electrons		
11	The water regain value is The amount of water required for gel swelling		
12	In the isolation of halide compounds by HPLC the best column used should made from Stainless steel lined with glass		
13	Chloromethylated polystyrene treated with 1ry amine give resin act at all pH value		
14	Velocity of Carrier Gas depend on the deference between inlet and outlet pressure		
15	We can add 4gm. Of dry gel grad 25 to concentrate 40 ml of protein solution from 25% to 50% solution		
16	Rubidium chloride is used for detection of phosphorus compounds.		

**Discuss why? .....(10 marks)**

1- Weak anionic exchange resins are not commonly found in the hydroxide form to act by its full capacity

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2- In GLC. The stationary phase should be applied as homogenous thin film coating the support

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3- The buffer is important in chromatographic separation

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4- Pyridine is used In silylation reaction

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5- Sephadex is Unstable at acidic pH

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6- The selection of the carrier gas is very important

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7- Adsorption forces play a greater role in sephadex LH20

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8- FID Not respond to CO<sub>2</sub>, CO, CHCO and CS<sub>2</sub>

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9- Ion exchange resins not used in HPLC

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10- Halide ions and prolonged use of organic buffers should be avoided in the mobile phase

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**Part (II): Quality control testing**

**20 marks**

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**A-Write short notes on the following, supporting your answer with examples and applications whenever applicable:**

**10 points**

**1-Analysis of dencichine as marker compound for quality control of ginseng:**

**2-Differential Pulse Polarography (DPP) in quality control of herbal drugs:**

3-Quantitative NMR (qNMR) and tandem mass spectroscopy as quality control techniques of herbal drugs:

4-HPLC assay of herbal drugs containing known compounds and herbal drugs containing several structurally related active principles:



5-Total, acid insoluble and water soluble ash as tools in assessing purity of herbal drugs

B-Match the following statements with the answers in table1 by writing the number of each statement in front of the correct answer: 10 points

- 1-Compounds analyzed by super critical fluid chromatography.
- 2-A technique, which uses which uses a polar stationary phase e.g. silica and a mobile phase containing appreciable amount of water and acetonitrile
- 3-A method for determination of number of starch grains per mg of sweet potato .
- 4-The percentage number of stomata per unit area of leaf epidermis.
- 5-An example of assessing purity and adulteration by HPLC.
- 6-A method for moisture determination in volatile oil containing drugs.
- 7-A parameter used in Purity assessment of volatile and fixed oils.
- 8-A mild ion generation technique in HPLC-MS of herbal drug extracts.
- 9-A type of PCR but the segments of DNA to be amplified are random with unknown target genome.
- 10-Quantitative chemical tests for volatile oils.

- 11-It is amplification strategy in which a PCR primer is designed to be able to discriminate among templates that differ by a single nucleotide residue ARMS-PCR
- 12-Active constituents of *Angelica sinensis* (Dong quai).
- 13-A technique used in quality control of *Stephania tetrandra*.
- 14-Active constituents of *Salvia miltiorrhiza* root (Lamiaceae).
- 15-Ailments treated by Ibrogast.
- 16-A drug used in GIT and respiratory tract ailments and in skin inflammations and burns.
- 17-It is a technique concerned with characterizing large numbers of metabolites using NMR, chromatography and mass spectrometry..
- 18-One use of X-Ray Powder Diffractometry
- 19-A technique used in study of different physical or chemical changes in herbal drugs.
- 20-It is the reciprocal of dilution of the drug still displaying bitter taste.

Table (1):

Answer	Number
Loss on drying	
Helium chryogenic techniques	
<i>Phytolacca dodecandra</i> saponins	
Stomatal index	
SIM2 primer	
Bis benzylisoquinoline alkaloids	
LC-APCI-MS	
HPTLC fingerprinting	
RFLP	
Refractive index	
Toluene distillation	
Marshmallow root	
Atmospheric pressure ionization	
Water-soluble phenolics and colored diterpene (s)	

Eye piece and stage micrometers	
Diversity in their 5s-rRNA gene	
Hydrphilic interaction chromatography (HILIC)	
Lycopodium_spore	
Nonaqueous capillary electrophoresis	
Optical rotation	
Elder flower	
Metabolomics	
ARMS-PCR	
Z-ligustilide	
Analysis of metallic based Indian traditional medicines	
nonvolatile or thermally labile and lacking functional groups	
Stomatal number	
Bitterness value	
Thermal analysis techniques (TGA, DTA, DSC)	
Acid value, ester value & saponification value	
High-speed countercurrent chromatography	
No need for the prior extraction and separation.	
Functional desepsia and irritable bowel syndrome	
RAPD	
Presence of <i>Parthenium integrifolium</i> in <i>Echinacea purpurea</i>	