BIRLA INSTITUTE OF TECHNOLOGY & SCIENCE
Pilani-333031. Rajasthan
Comprehensive Examination (First Semester 2023-2024)

Course Name: Modern Pharmaceutical Analytical Techniques Course No: PHA G540
Total Marks: 35 Date: 12-12-2023 Duration: 180 (min)

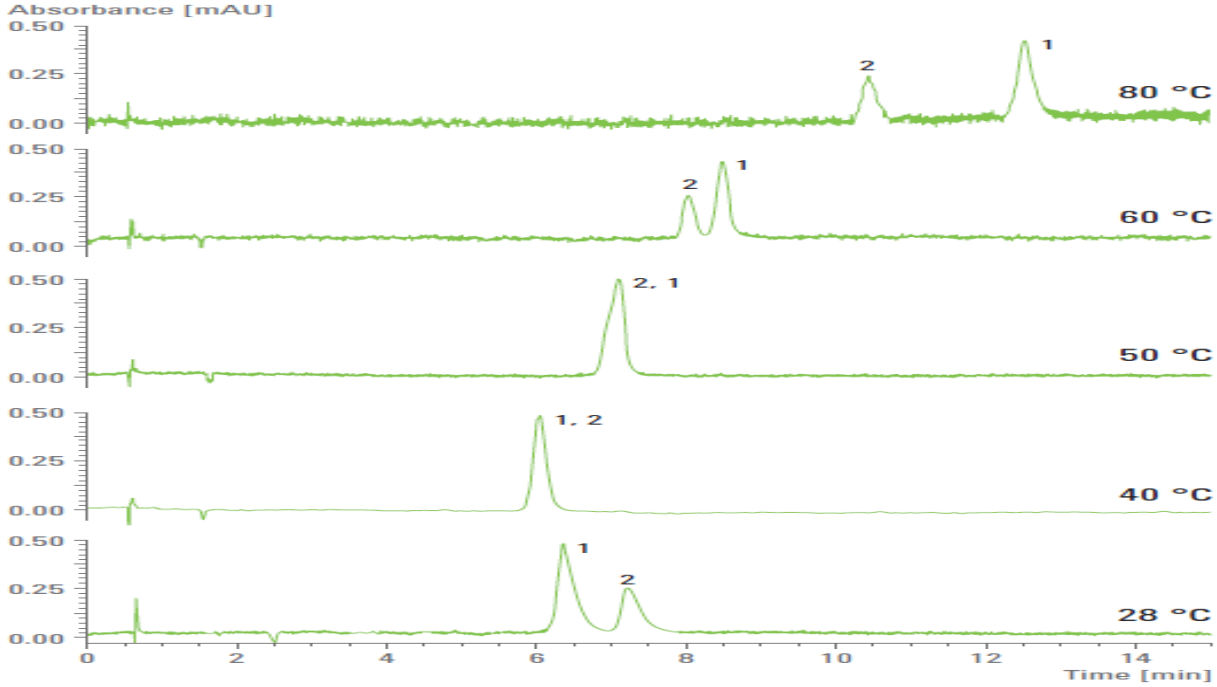
Note: Answer for all questions precisely with appropriate illustrations if required.
Give the answer for part-A and part-B separately.
Give the answer for all sub-parts together in one place.

Part-A (Closed Book) **5x2=10 Marks**

1) How will you check linearity and accuracy of a newly developed analytical method as an analyst in a MNC, explain?

2) Write a note on LC technique used for separation and purification of bio-molecule with respect to their individual chemical structure.

3) Comment on the following chromatogram obtained for sample 1 and 2,



4) How will you analyze your sample using Pyrolysis GC and Headspace GC?

5) Write the construction and working principle of TQMS.

Part-B (Open Book) **25 Marks**

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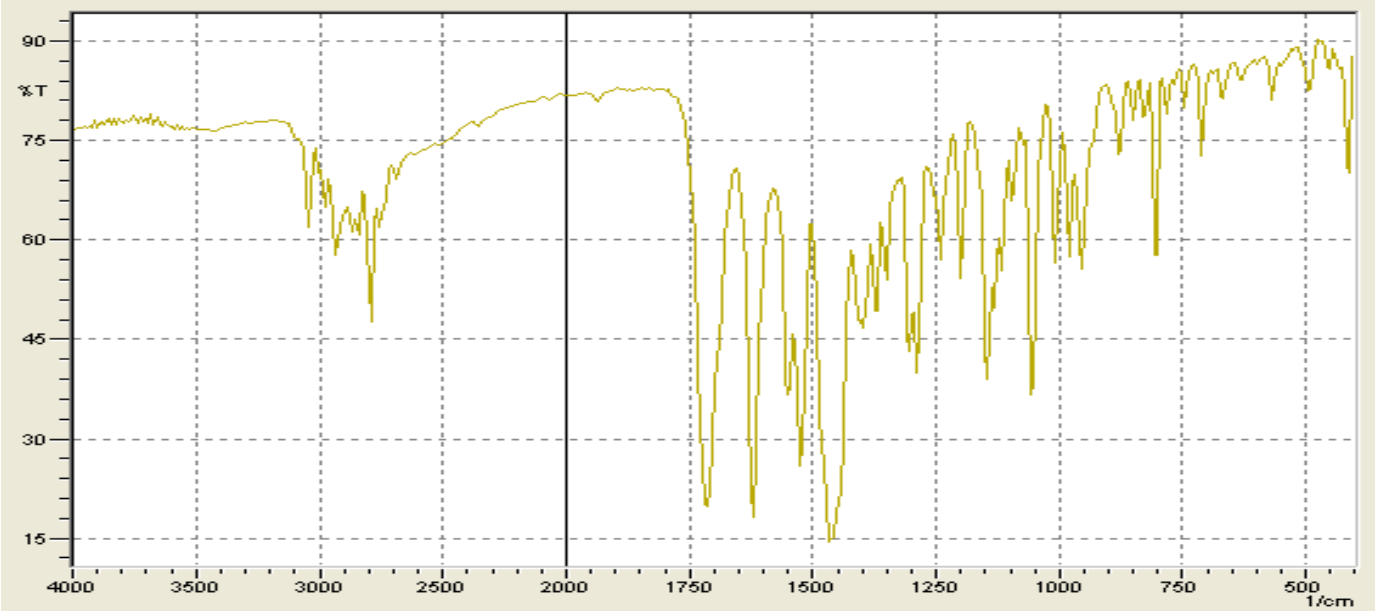
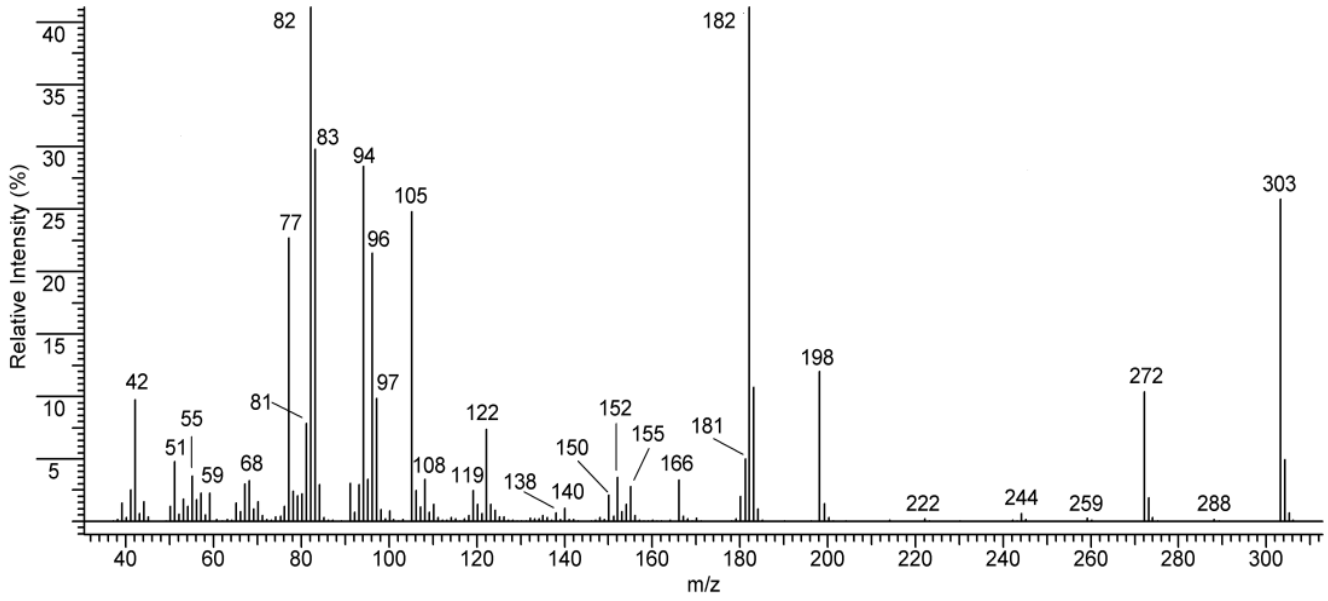
1) Interpret the following spectrum of given sample (Molecular formula $C_{18}H_{20}FN_3O_4$) and report the details of the sample as well as possible structure if any,

(2)



2) a) Interpret the following spectrum of compound (Mol. for: $C_{17}H_{21}NO_4$) and write your inference. **(2x2-**



4)

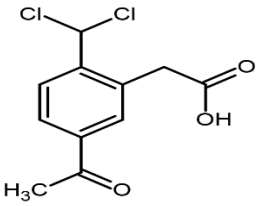
b) Predict the number of Carbon, Nitrogen and Hydrogen atoms present in the following compound whose mass spectral data as follows by applying all possible rules,

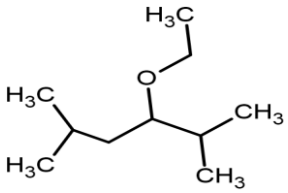
m/z = 121 (M; 47.9%), M+1 = 122 (4%)

3) For each molecule, predict the sets of non-equivalent H's present, number of signals in the 1H-NMR, relative intensity of signals and splitting pattern of each proton.

(2x2=4)

a) b)





4) a) Assign the elution order for the following analytes with justification. The analytical conditions are as follows,

(4x2=8)

Column : YMC-Pack CN (5 µm, 120 Å), 250×4.6 mm I.D

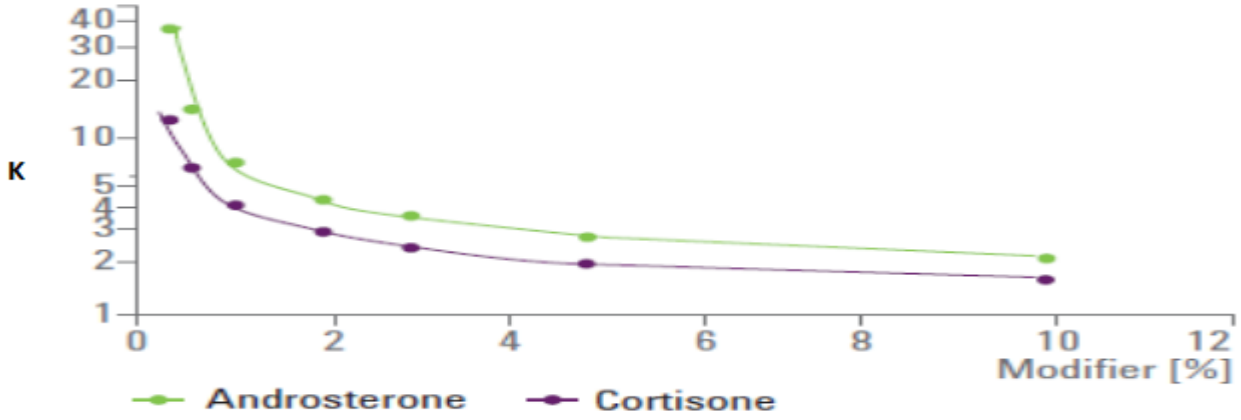
Eluent : acetonitrile/water (40/60); Flow rate : 0.6 mL/min; Pressure : 5.6 MPa; Temperature : 30 ℃

Detection : UV at 254 nm, 0.32 AUFS; Injection : 3 µL (0.25 mg/mL)

i) Corticosterone, Deoxy-corticosterone, Progesterone, Hydrocortisone

ii) What will happen, if eluent has been changed to hexane/methanol/dichloromethane (75/20/5) and other parameters remain unchanged.

b) Below is the chromatogram obtained during SCFC of steroids androsterone and cortisone. Write your inference regarding the relationship between the plotted parameters.



c) Write the inference for the given summary of results obtained during the development of HPTLC methods for Carbamazepine (CBZ) sample.

Parameters	Amount of CBZ spotted (ng)	Amount of CBZ detected (ng, mean ± SD)	%RSD
Mobile phase composition: 5.1 : 3.9 : 1.0	300	298.23 ± 1.7	2.00
Mobile phase composition: 6.9 : 4.1 : 1.0	300	301.1 ± 1.35	2.30
Mobile phase volume: 8 mL	300	297.7 ± 2.06	1.10
Mobile phase volume: 12 mL	300	295.44 ± 4.24	2.60
Chamber saturation time: 15 min	300	298.9 ± 1.41	1.90

Chamber saturation time: 25 min	300	300.6 ± 1.23	2.00
Solvent migration distance: 68 mm	300	297.7 ± 3.07	1.60
Solvent migration distance: 72 mm	300	299.04 ± 1.9	1.70

d) A GC column was operated under the following conditions:
Column: 1.10 m x 2.0 mm, packed with Chromosorb P;
weight of stationary liquid added, 1,40 g; density of liquid, 1.02 g/mL
measured outlet flow rate: 5.3 mL/min
temperature: room, 21.2°C; column, 102.0°C
retention times: air, 18,0 s; methyl acetate, 1.98 min; methyl propionate, 4.16 min; methyl n-butyrate, 7.93 min
peak widths of esters at base: 0.19, 0.39 and 0.79, respectively
Calculate the following,
(i) The average number of theoretical plates and plate height for the column.
(ii) The resolution for each adjacent pair of compounds.

5) a) Determine the empirical formula and molecular formula for the given elemental data. The molecular weight of this compound is 366 g/mol.
(2x2=4)
81.97% carbon, 6.01% hydrogen, 7.65% nitrogen and 4.37% of oxygen

b) Calculate the % of C, H, N and O for the following compound with molecular formula of $C_{26}H_{24}N_2O_2$.

6) a) What is the enantiomeric excess of a solution with a specific rotation of –50 where the pure solution rotates at –150? For the above same solution, how much of the (–) and (+) enantiomers are present? **(2x1.5=3)**

b) 2-butanol has a specific rotation of +58.26^o. How many grams of 2-butanol is required to produce an angle of rotation of 44^o using a 10 mL cell with a path length of 5 cm?
