BIRLA INSTITUTE OF TECHNOLOGY & SCIENCE
Pilani-333031. Rajasthan
Second Semester 2022-2023
Comprehensive Examination

Course Name: Modern Pharmaceutical Analytical Techniques Course No: PHA G540
Total Marks: 30 Date: 09-05-2023 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) Write a note on common types of errors in an instrumental methods of analysis and how will you overcome the same.

2) Write a roadmap of the most appropriate analytical method for the determination and quantification of residual solvents in paclitaxel.

3) How will you classify chromatography based on attractive forces of separation? Enumerate a note on high-resolution medium pressure bio-chromatography?

4) Write a brief account on RPC and OPLC technique.

5) Which is the preferred greener analytical method for the separation and determination of non-volatile, thermally labile compounds that are not conveniently handled by either GC or LC. Enumerate the procedure for the same.

Part-B (Open Book) **20 Marks**

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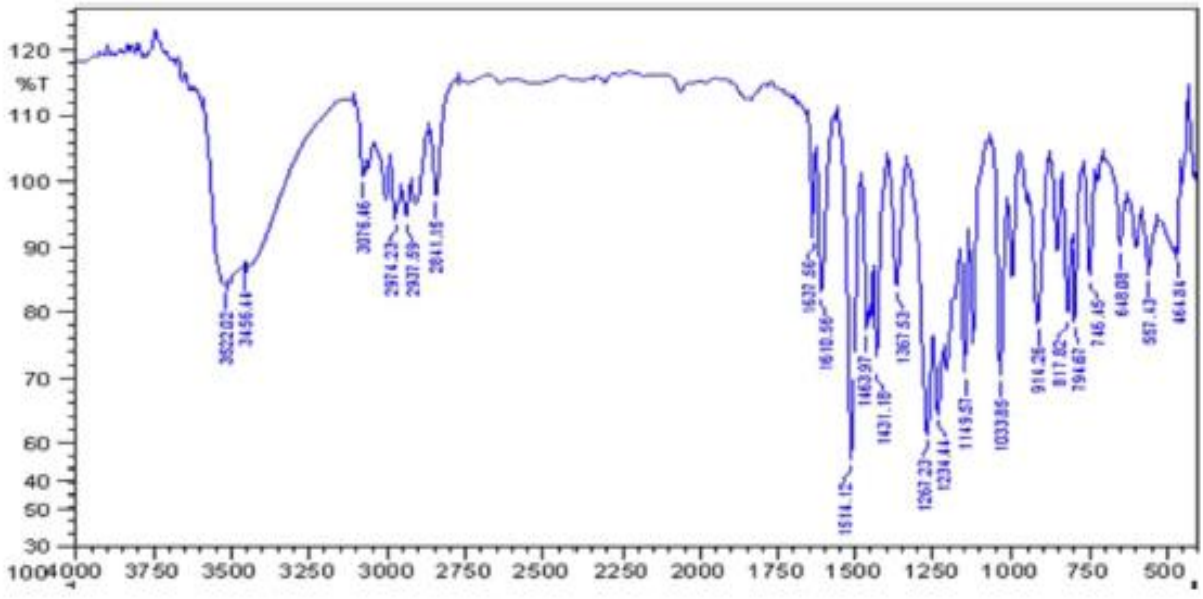
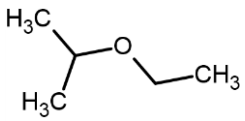
1) Interpret the following IR spectrum of given sample (Molecular formula $C_{10}H_{12}O_2$) and report the details of the sample as well as possible structure if any,
(6x3=18)



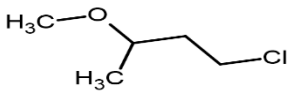
2) Deduce the suitable molecular formula that corresponds to the mass spectral data by applying all possible rules.
m/z = 150 (M; 100%), M+1 = 151 (10.20%) and M+2 = 152 (0.88 %). Assume presence of Oxygen in this compound.

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.

a)



b)



4) Write the inference for the given summary of results obtained during the development of RP-HPLC method for ledipasvir.

Factor[b]	Level	[c]t_R	[d]K	[e]T
Flow Rate (mL/min)				
0.9	-1	6	3.80	0.15
1.0	0	6.1	3.13	0.21
1.1	+1	6	2.85	0.20
Mean ± S.D.		6.03 +/- 0.05	3.26 +/- 0.49	0.19 +/- 0.03
% Acetonitrile in the mobile phase (v/v)				
49	-1	6	3.58	0.39
50	0	6.2	3.63	0.48
51	+1	6	3.43	0.53
Mean ± S.D.		6.06 +/- 0.11	3.55 +/- 0.08	0.47 +/- 0.06

[a]Average of three concentrations 20 µg/mL
[b]Two factors were slightly changed at three levels (-1, 0 and +1)
[c]t_R= retention time (min)
[d]K= retention factor
[e]T= tailing factor

5) 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: 25.3 mL/min
temperature: room, 21.2°C; column, 102.0°C
retention times: air=0.3 min; methyl acetate=1.98 min; methyl propionate=4.16 min; methyl n-butyrate=7.93 min
Calculate
(i) the retention factor for each component.
(ii) selectivity factor for each adjacent pair of compounds.

6) Determine the empirical formula and molecular formula for the given elemental data. The molecular weight of this compound is 146.10 g/mol.
57.54% C, 3.45% H and 39.01% F.

7) A 17 g sample of impure Ibuprofen was dissolved in 100 ml of water and it's optical rotation was determined using 10 cm cell at 25 ^{o} C. If the observed value of rotation is -2.38 ^{o}. Calculate the percent by weight (% purity) of Ibuprofen in the sample. Specific rotation of Ibuprofen is -14.49 ^{o}.
(2 M)
