BIRLA INSTITUTE OF TECHNOLOGY & SCIENCE, PILANI
Second Semester 2021-2022
Comprehensive Examination

Course Name: Modern Pharmaceutical Analytical Techniques Course No: PHA G540
Total Marks: 30 Date: 20-05-2022 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

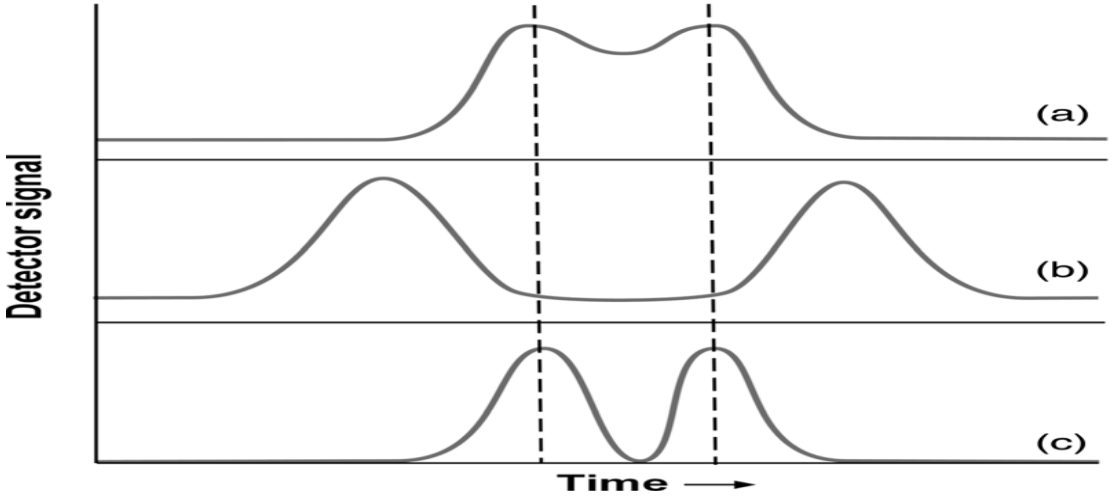
1) How will you prepare / pre-treat the given sample of empagliflozin tablet for appropriate instrumental analysis and estimate the content of the same indirectly.

2) Write the roadmap for the analytical method development of given non-polar compounds using HPTLC.

3) Write a roadmap of the most appropriate analytical method for the determination and quantification of residual solvents of Amyl nitrite sample.

4) 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.

5) Comment on the following chromatogram with justification, assume column, flow rate and temperature are constant.



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

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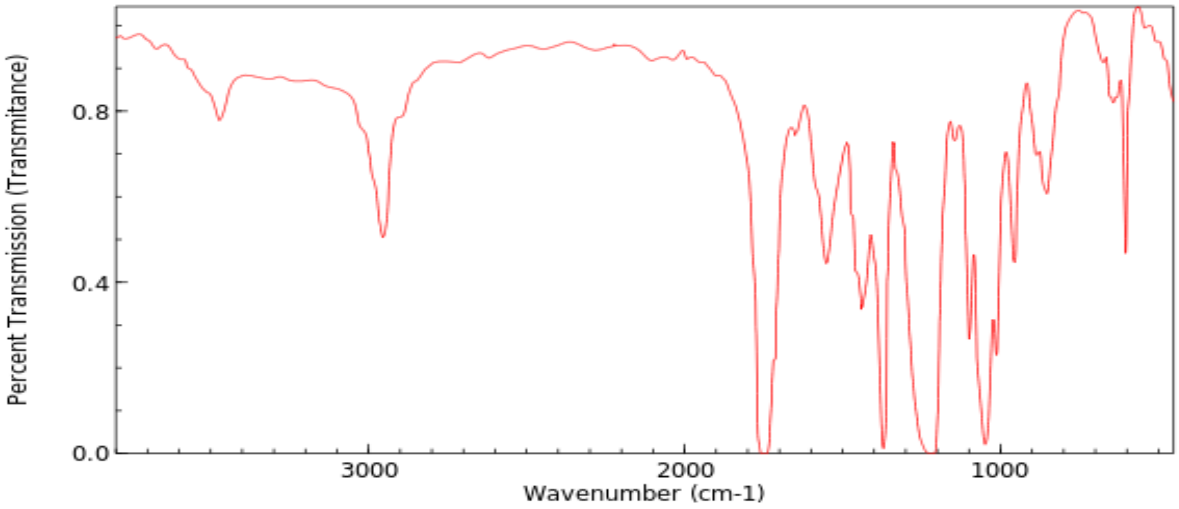
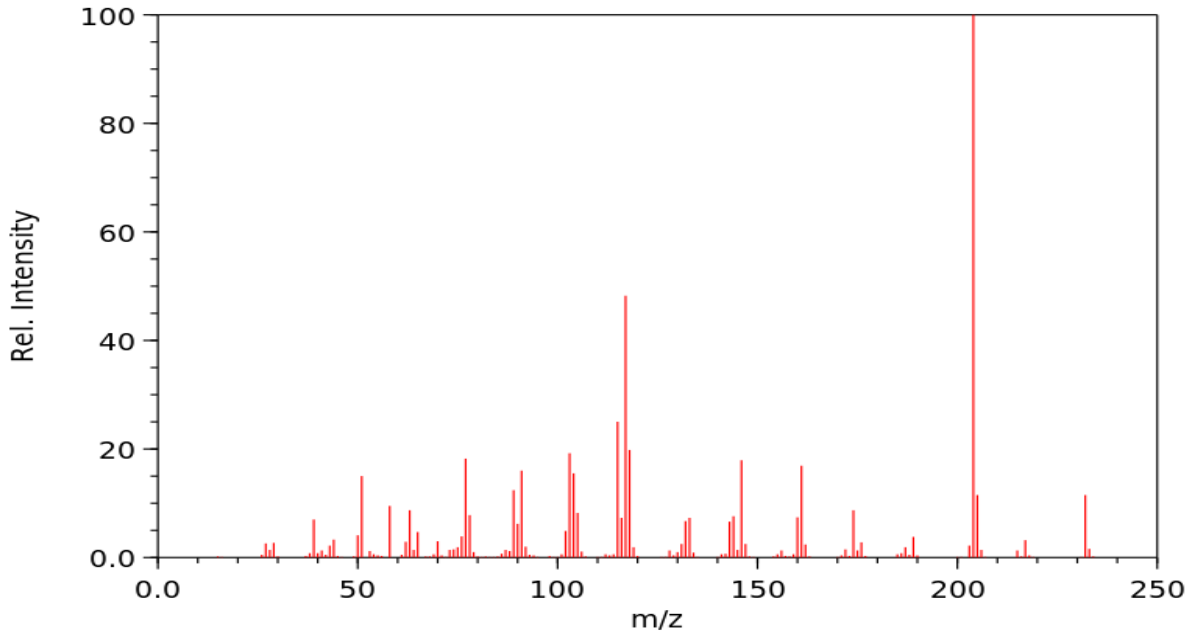
1) Interpret the following IR spectrum of given sample (Molecular formula $C_9H_{14}O_6$) and report the details of the sample as well as possible structure if any, **(3)**



2) Interpret the following Mass spectrum of the given sample (Mol. formula: $C_{12}H_{12}N_2O_3$) and write your inference. **(3)**

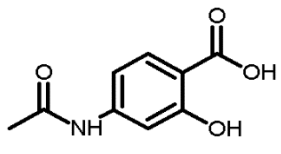


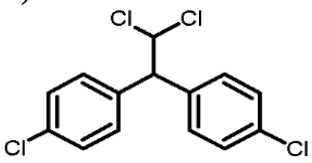
3) Deduce the molecular formula that corresponds to the mass spectral data by applying all possible rules. **(3)**

m/z = 220 (M; 100%), M+1= 221 (9.43%), M+2= 222 (0.4%)

4) For each given 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. **(2x1.5=3)**

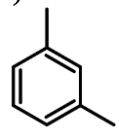
a) b)

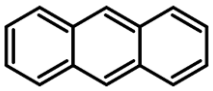




5) Predict the sets of equivalent C's and number of C-NMR signals for the following. **(2x1.5=3)**

a) b)



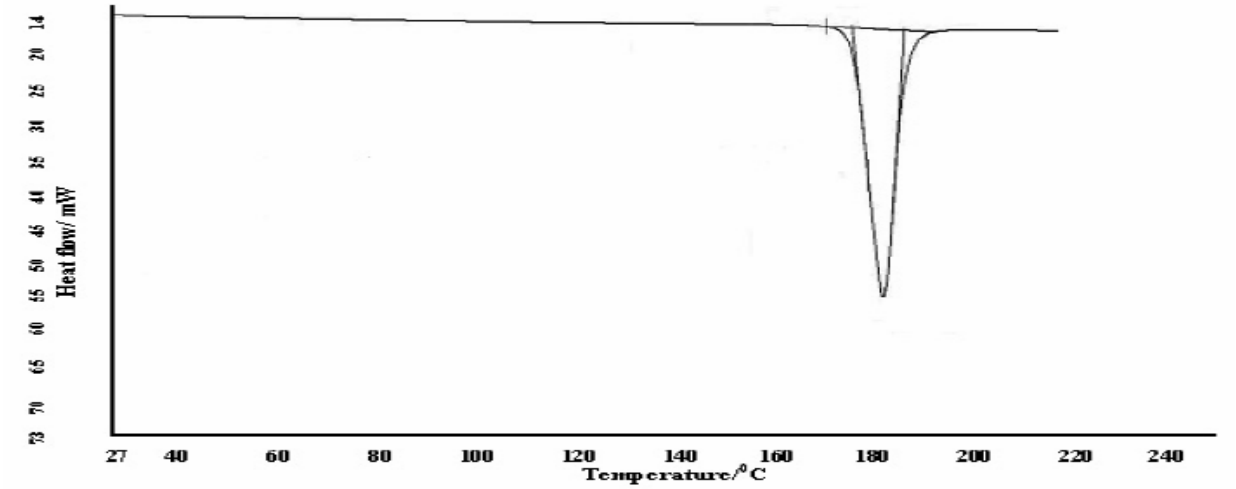


6) a) Determine the empirical formula and molecular formula for the given elemental data. The molecular weight of this compound is 284 g/mol. **(2x1.5=3)**

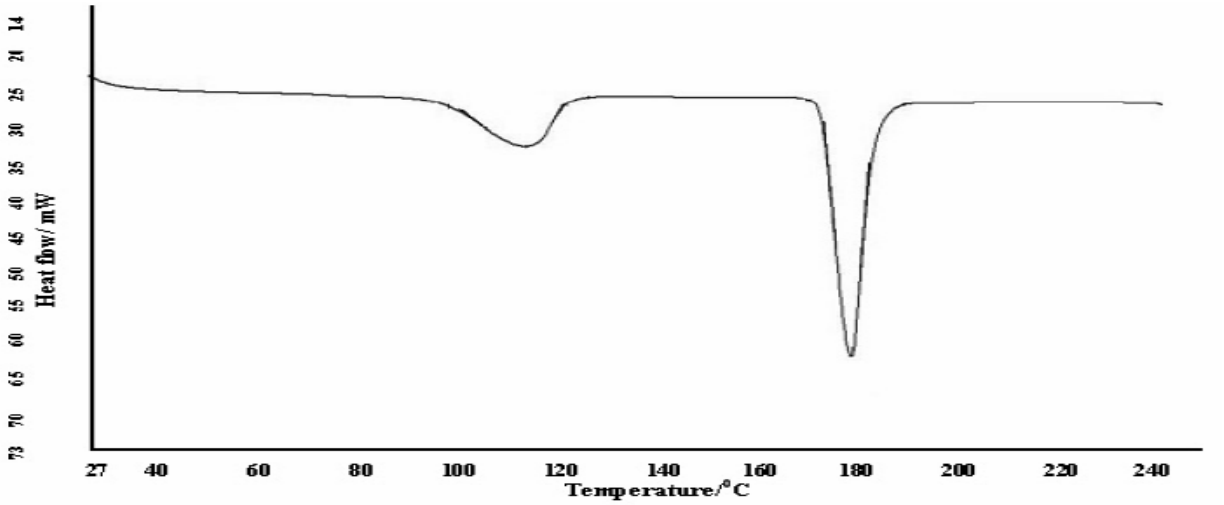
C, 59.14; H, 7.09; O, 33.77

b) Calculate the % of C, H and O for the following compound with molecular formula of $C_{20}H_{14}O_4$

7) Interpret the following thermograph and write your inference and discussions. **(2)**



DSC thermogram of pure stavudine



DSC thermogram of stavudine and magnesium stearate
