

**BIRLA INSTITUTE OF TECHNOLOGY & SCIENCE, PILANI**

**First Semester 2022-2023**

**Comprehensive Examination**

**Course Name: Modern Pharmaceutical Analytical Techniques Course No: PHA G540**

**Total Marks: 35**

**Date: 17-12-2022**

**Duration: 180 (min)**

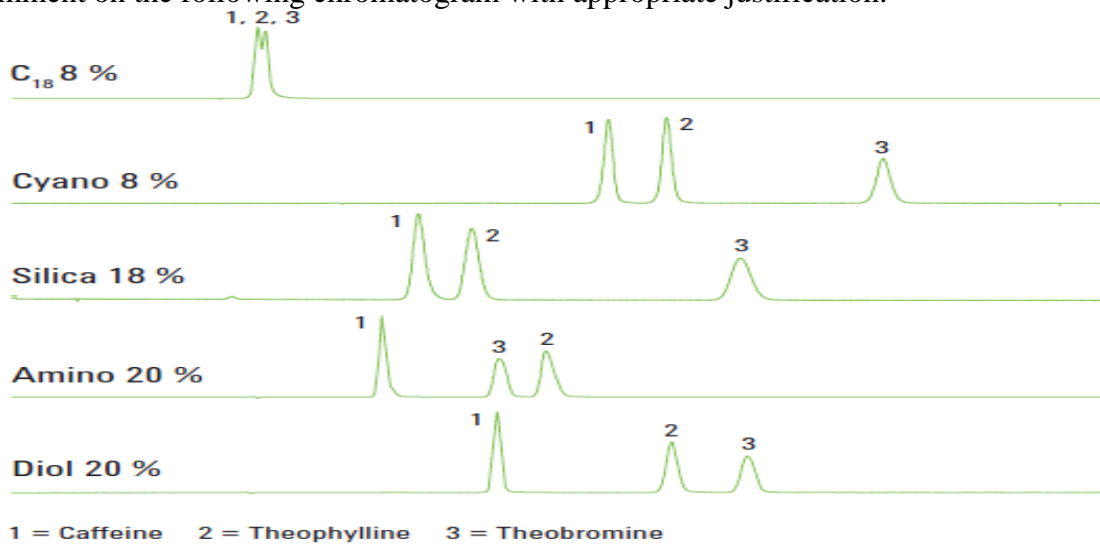
**Note: Answer for all questions precisely with appropriate illustrations if required.**

**Give the answer for part-A and part-B separately.**

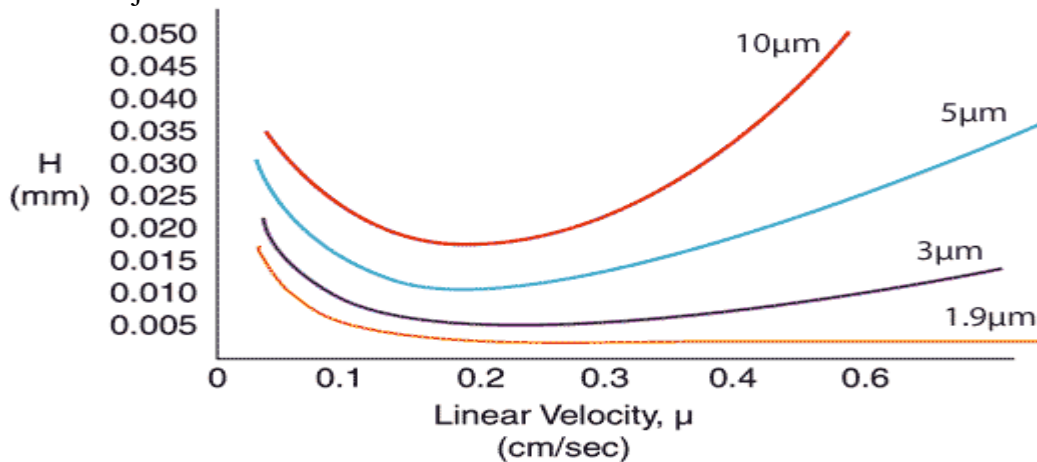
**Part-A (Closed Book)**

**5x2=10 Marks**

- 1) Write the principle and steps involved in SPE method.
- 2) How will you classify chromatography based on the mobile phases used? Enumerate the steps involved in AC?
- 3) Write a brief account on grafted TLC and SRS technique.
- 4) Comment on the following chromatogram with appropriate justification.



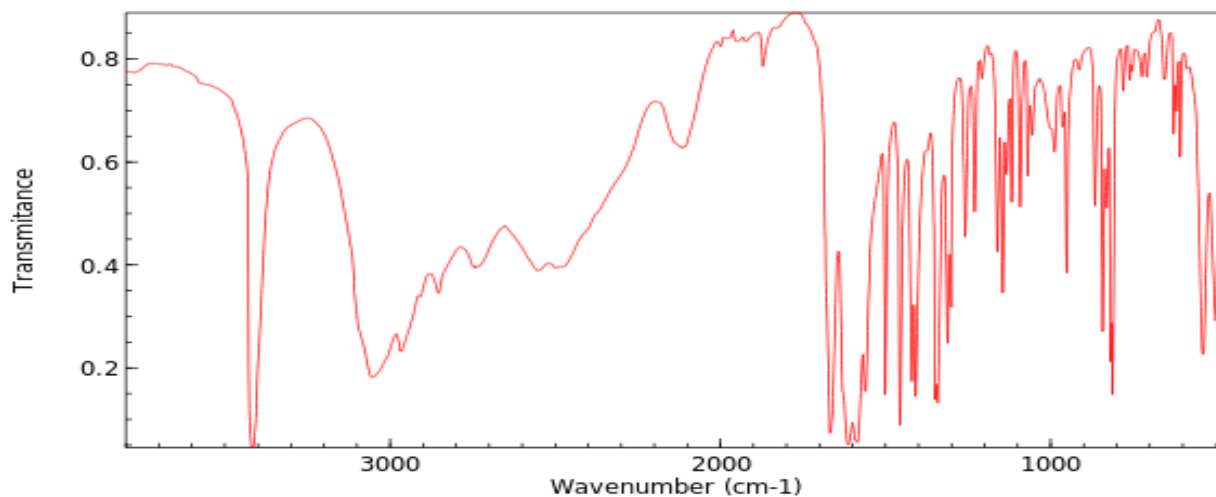
- 5) Interpret the following graph between plate height Vs flow velocity upon varied column particle size with justification.



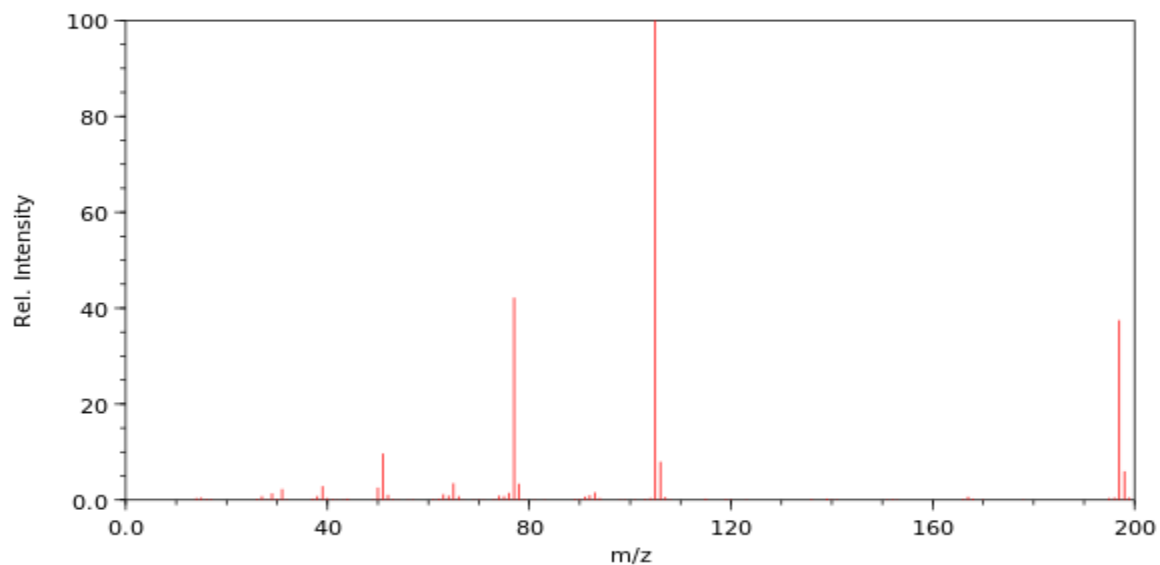
**Part-B (Open Book)**

**25 Marks**

1) Interpret the following IR spectrum of given sample (Molecular formula  $C_{11}H_{11}FN_2O_2$ ) and report the details of the sample (aliphatic / aromatic, DBE, saturated / unsaturated, functional group) as well as possible structure if any, (3)



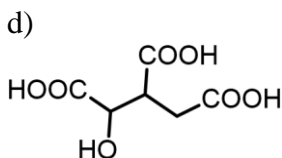
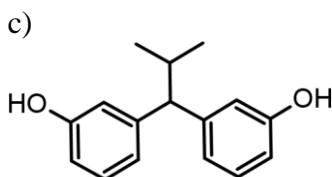
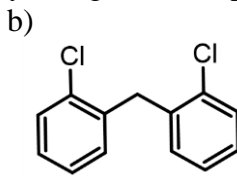
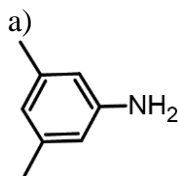
2) a) Interpret the following Mass spectrum of the given sample (Mol. formula:  $C_{13}H_{11}NO$ ) and write your inference (base peak, parent peak, satellite peak, fragment peaks) (2x3=6)



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

$m/z = 142$  (M; 100%),  $M+1=143$  (8.9%),  $M+2= 144$  (0.26%). Assume absence of Oxygen in this compound.

3) For each given molecule, predict the sets of non-equivalent H's present, number of signals in the  $^1\text{H-NMR}$ , relative intensity of signals and splitting pattern of each proton. **(4x1.5=6)**



4) a) Determine the empirical formula and molecular formula for the given elemental data. The molecular weight of this compound is 102.2 g/mol. **(2x3=6)**  
S, 31.42 %; O, 31.35 % and F, 37.23 %

b) You are provided with sample composed of three compounds A, B and C with different polarities. The sample is loaded onto a C18 column and eluted with 30% methanol/water mobile phase. After eluting with 10 mL of solvent and collecting 1 mL fractions, compounds A and B are found to be in the 3rd and 6th fractions, respectively.

(i) Which compound is more polar, A or B? Justify

(ii) After eluting with another 10 mL of 30% methanol/water mobile phase, compound C has still not eluted. Describe how will you obtain compound C.

(iii) Is compound C more or less polar than compounds A & B? Justify

5) a) The (+) enantiomer of an optically active compound has specific rotation ( $[\alpha]_{20D}$ ) of  $+100^\circ$ . For a sample (1 g/ml in 10 cm cell) that is a mixture of (+) and (-) enantiomers, the observed rotation  $\alpha$  is  $-45^\circ$ , what is the percentage of (+) enantiomer present in that sample? **(2x2=4)**

b) Interpret the following and write your inference and discussions.

