27-11-12

Date: 27/11/12 **Exam no. _____**

Maliba Pharmacy College
Uka Tarsadia University
Mid-Semester Examination
M. Pharm. (1st Semester)
Modern Analytical Techniques

Duration: 3 hours **Maximum marks: 70**

Q.1]

(a) Identify the following compounds on the basis of the spectral data given below. Show the reasoning for the conclusion arrived at. **(Any Two)** **[8]**

i. M.F $C_4H_5O_2N$
IR: 2250, 1740, 1240, cm^{-1}
NMR: (δ) 3.8 s (3H), 3.5 s (2H)

ii. IR: 3030, 2900, 1600, 1550, 750, 690 cm^{-1}
NMR: (δ) 0.7 t (3H), 1.3 s (6H), 1.7 quartet (2H), 7.2 s (5H)
MS: M^+ 148, 119, 105, 91, 77.

iii UV: 270 nm (€ = 1450)
IR: 3500, 3000, 1612, 1595, 1507, 1223, 813 cm^{-1}
NMR: (δ) 2.3 s (3H), 5.1 s (1H), 7.1 s (2H), 8.2 s (2H)
MS: 108, 107, 79, 77, 51.

(b) Give chemical shift values and spin coupling for following compounds **[4]**

i. Acetaldehyde ii. Ethyl bromide
iii. Ethyl methyl ether iv. 2-chloro propane

(c) TMS is used as reference compound in NMR. Explain. **[2]**

Q.2]

(a) **Attempt any two** **[10]**

i. Describe the instrumentation and applications of DSC.
ii. Discuss the theories of chromatography.
iii. Discuss hydrogen decoupling and off resonance decoupling in CMR spectroscopy.

(b) **Define the following terms** **[4]**

i. Capacity factor ii. Reversed phase chromatography
iii. Partition ratio iv. Solvent programming

Q.3]

(a) **Attempt any two** **[14]**

i. What is chemical shift? Describe the factors affecting chemical shift.

ii. What is time and frequency domain? Describe the principle and working of Michelson interferometer with diagram.

iii. Explain the theory of size exclusion chromatography. State its applications. Describe the stationary phase in SEC.

Q.4]

(a) **Answer the following** **[6]**

i. Explain working of Hollow cathode lamp and Electrode less discharge lamp with suitable diagrams.

ii. What is plasma? Explain working of ICP torch with suitable diagram. State limitations of ICP torch over DCP.

(b) **Explain the following statements (any four)** **[8]**

i. On hydrogen bonding stretching frequency in IR gets lowered.

ii. M+ is not obtained for sugars using electron impact source.

iii. Improvement in S/N ratio is observed with FT-IR.

iv. Anilinium cation exhibits UV spectrum almost identical to benzene.

v. Mono substituted alkyl benzene shows prominent peak in MS.

Q.5]

(a) Enumerate various types of mass analyser. Describe quadrupole mass anlyser with appropriate diagram. **[4]**

(b) **In a reversed phase chromatography, substances A and B have retention times of 18.34 and 19.75 min., respectively, on a 30.0 cm column. An unretained species passes through the column in 1.35 min. The peak widths (at base) for A and B are 1.16 and 1.28 min., respectively. Calculate column efficiency and column resolution. In what possible ways can you improve the resolution to 1.5? Which option would you prefer? (Give reason and show calculation). Mobile phase used for separation of A and B was 43% (by volume) methanol and 57% water. Calculate a water-methanol composition that should bring capacity factor of B to a value of about 4.**

[10]