

STJOSEPH'S COLLEGE(AUTONOMOUS), BANGALURU

**M.Sc. CHEMISTRY-III SEMESTER
MID SEMESTER TEST – AUGUST 2016**

CH 9210: SEPARATION AND ELECTROANALYTICAL CHEMISTRY

Time: 90 min

Max. Marks: 35

Part A

Answer ANY THREE questions from this part. Each question carries 2 marks. (3x2=6)

1. Define $pH_{1/2}$. What should be the minimum $pH_{1/2}$ difference between two divalent metal ions to achieve a separation with 1% cross contamination?
2. The distribution coefficient for X between CCl_4 and water is 9.6. If 50ml of an aqueous solution of 0.150M X is contacted with 40ml of CCl_4 , calculate the concentration of X remaining in water.
3. In a chromatographic analysis of lemon oil a peak for limonene has a retention time of 8.36 min with a baseline width of 0.96min. γ -terpinene elutes at 9.54min with a baseline width of 0.64min. Calculate the resolution.
4. Explain how is the direction of electroosmotic flow reversed in capillary electrophoresis?
5. With an example explain metallic indicator electrode of the second kind.

Part B

Answer ANY TWO questions. Each question carries 12 marks. (2x12=24)

6. Answer any four of the following questions based on the components of gas chromatogram: a) Draw the block diagram of the gas chromatograph b) Compare between narrow bore column with thin layer stationary phase and narrow bore column with thick layer stationary phase c) How does soap bubble flowmeter work? d) How does Flame ionization detector work? e) Explain the split and splitless injection port f) Give one example each for the polar stationary phase and non polar stationary phase.
7. a) Substances A and B have retention times 16.40 and 17.65 minutes respectively on a 30m column. An unretained species passes through the column in 1.30 minutes. The peak width (at the base) for A and B are 1.11 and 1.21min respectively. Calculate the i] average number of plates for A and B c] average plate height ii] the length of the column required to achieve the baseline separation iii] retardation factor for both A and B iv] selectivity factor. b) Explain the following terms (i) Column bleed (ii) Septum purge. (8+4)
8. (a) With the help of a schematic diagram discuss the electrophoretic mobility and order of separation of the solutes in the case of capillary zone electrophoresis
(b) With relevant equations discuss the electrical conductance across the glass membrane.
(c) Discuss the separation of nitro compounds using micellar electrokinetic capillary chromatography technique

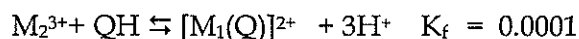
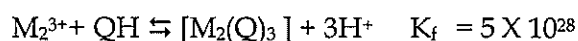
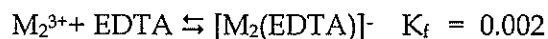
[5+3+4]

Part C

Answer **ANY ONE** question. Each question carries **5 marks**.

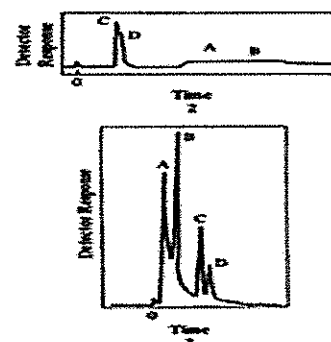
(1x5=5)

9. a) The K_f for two reactions are given below:



Where QH is 8-hydroxy quinoline and EDTA is ethylene diamine tetra acetate. Design a separation method to separate M1 and M2 with proper explanation.

b). Four analytes A, B, C and D are injected in a column with silica as the stationary phase. First chromatogram (1) was the result. The column was treated and again the separation was done. Chromatogram 2 was obtained. Explain (i) The order of elution in 1 and 2. (ii) The shape of peaks in 1 and 2 (iii) what treatment might have been done in the column material? (2+3)



A = EtOH
B = methylethyl ketone
C = benzene
D = cyclohexane

10. Diet soft drinks contain appreciable quantities of riboflavin (a neutral compound), aspartame, benzoic acid and caffeine. With relevant explanation predict the expected order of elution for these compounds in a capillary zone electrophoresis separation using a pH 9.4 buffer solution, given that aspartame has pKa value of 2.964; benzoic acid pKa is 4.2 and pKa of caffeine is less than zero.