THE OPEN UNIVERSITY OF SRILANKA
CHU 3129 – INSTRUMENTAL METHODS OF CHEMICAL ANALYSIS

Answer Guide – Assignment Test 3

1.(i) In reverse phase chromatography stationary phase is non polar and mobile phase is polar. Polar compounds are more attracted to polar solvents.

(ii) Any solvents but the mobile phase should be polar than the stationary phase (Refer Unit III page number 17).

(iii) When a separated band or zone moves down a column it tends to spread or become broader and overlap with other zones. This is called band broadening.
Write any two reason among the following.
Slow linear flow rate, eddy diffusion, longitudinal diffusion, low diffusion coefficient in stationary phase, high resistant to mass transfer in the mobile phase

(iv) In electrophoresis there is no mobile phase.

(v) The size of the molecule – when the size increases rate of migration of ions decreases.
Net charge – when charge increases the rate of migration increases.
Strength of the electric field – when strength of the electric field increases rate of migration increases.

2.(i) Gas solid chromatography is usually used to separate gases and very volatile compounds with low molecular weight. They have short retention times with the most widely used stationary phases. Otherwise the retention time will be very high and tailing will occur.

(ii) Write any 3 properties in the following.
Should be thermally stable.
Should have negligible volatility.
Should be chemically inert.
Should have high selectivity factor.
Should have high retention factor.

(iii) The detectors used in GC do not respond exactly the same way to every compound. We can correct this error by using the relative response factor.

(iv) The compounds that are retained less will elute at a low temperature while compounds that are strongly retained will elute at a high temperature; resolution will not be lost while completing the separation in a reasonable time.

(v) a) The units of width should be corrected as 4.00 min (therefore we did not mark The calculation, however the marks were given for the equation)
$N = 16t_a^2 / w^2$
If w = 4.00 min $N = 16 \times 15^2 / 4^2 = 225$

b) $$k'_A = \frac{t_A - t_m}{t_m}$$

$$5 = (15 - t_m)/t_m \qquad t_m = 2.5 \text{ min } (150 \text{ seconds})$$

c) substituting $X = 1.5$, $t_A = 15$ min $t_m = 2.5$ min

$$X = \frac{t_B - t_m}{t_A - t_m} \quad \text{or} \quad X = \frac{t_A - t_m}{t_B - t_m}$$

$$= 21.25 \text{ min } (1275 \text{ sec}) \qquad = 10.83 \text{ min } (649.8 \text{ sec})$$

3. (i) $Cd_{(s)} / Cd(NO_3)_{2\,(aq)} \,//\, KCl_{(aq)} / AgCl_{(s)} / Ag_{(s)}$

(ii) $Cd_{(s)} \rightarrow Cd^{2+}_{(aq)} + 2e$
$AgCl_{(s)} + e \rightarrow Ag_{(s)} + Cl^-_{(aq)}$

$Cd_{(s)} + 2\,AgCl_{(s)} \rightarrow Cd^{2+}_{(aq)} + 2\,Ag_{(s)} + 2\,Cl^-_{(aq)}$

(iii) $E = E^0 - \frac{RT}{nF} \ln \frac{[Products]}{[Reactants]}$

$E = (E^0_{cathode} - E^0_{anode}) - \frac{RT}{2F} \ln [Cl^-_{(aq)}]^2 [Cd^{2+}_{(aq)}]$

$E = (E^0_{Agcl/Ag} - E^0_{cd^{2+}/cd}) - \frac{RT}{2F} \ln [Cl^-_{(aq)}]^2 [Cd^{2+}_{(aq)}]$

$E = (0.222 - (-0.402)) - \frac{0.0592}{2} \log (0.50)^2 [Cd^{2+}_{(aq)}]$

$-2.601 = \log \{0.25x\, [Cd^{2+}_{(aq)}]\}$

$10^{-2.601} = 0.25 \times [Cd^{2+}_{(aq)}]$

$[Cd^{2+}_{(aq)}] = 0.01 \text{ mol dm}^{-3}$

(iv) Coulometry – measuring current as a function of time
Voltametry - measuring current as a function of applied voltage

(v) In polarography,
We use a special working electrode called dropping mercury electrode; unlike other types of voltametry ,the solution is not stirred.

(vi) More accurate (can determine equivalence point using large number of data); Can use for turbid or colored solutions where indicators can not be used; More sensitive