19.

S U M M A R Y :

SUMMARY

1. Ultraviolet absorption spectra of coumarins : (1)

Ultraviolet absorption spectra of coumarin and of methyl-, methoxy- and hydroxycoumarins have been studied in methanol in the range 240-360 mμ. The absorption maxima are essentially same for hydroxy- and methoxycoumarins, if the substituent is in 5,6,7 or 8 position ; however, the positions 5 or 8, 6 or 7 can be distinguished. It is possible to distinguish isomeric monomethyl- and dimethylcoumarins from a study of their ultraviolet absorption data.

2. Sorption of coumarins (2) :

The column sorption-desorption of coumarin with water and methanol as solvents and the comparative sorption of coumarin and methoxy-, methyl- and hydroxycoumarins in 10 % methanol (by volume) on the chloride form of the strongly basic anion exchange resin, Amberlite IRA-400, have been studied. The sorption increases in the order ; coumarin < methoxycoumarins < methylcoumarins < hydroxycoumarins. For methoxycoumarins the sorption study indicates that sorption increases in the order ; dimethoxy < monomethoxy and 8-methoxy < 6-methoxy. On the other hand, the sorption for hydroxycoumarins increases in the order ; monohydroxy < dihydroxy and 6-hydroxy < 8-hydroxy < 7-hydroxy < 5-hydroxy.

3. Separation of coumarins (3) :

The column separation of coumarins has been studied with a strongly basic anion exchange resin Amberlite IRA-400

in the chloride form. The coumarins studied are of three groups (A) includes coumarin, 3-Me(I), 7-Me(I), 3,4-diMe(I), 6,7-diMeO-4Me(I) and 7,8-diMeO(I) ; group (B) includes 7-OH(I) and 8-OH(I) ; group(C) includes 6,7-diOH-4Me(I) and 7,8-diOH-4Me(I). (I) denotes coumarin . Group (A) compounds were eluted by 10 % methanol, Group (B) compounds were not eluted by 10 % methanol but were slowly eluted by N/10 HCl in 10 % methanol. Group (C) compounds were not eluted by 10 % methanol or by N/10 HCl in 10 % methanol but were eluted by N/100 HCl in methanol. It is therefore possible to separate binary and ternary mixtures containing not more than one compound from each group.

4. Ultraviolet absorption spectra of cinchona alkaloid sulphates (4) :

Ultraviolet absorption studies have been made for aqueous solutions of cinchona alkaloid sulphates, (quinidine and cinchonine) of different pH (adjusted by addition of sulphuric acid or sodium hydroxide) and the ultraviolet absorption at invarient wavelengths has been used for the estimation of the alkaloid sulphate in dilute aqueous solutions.

5. Selectivity coefficients of sulphonic acid cation exchange resin in sulphate and chloride solutions (5) :

The exchange equilibria with a styrene divinylbenzene copolymer based sulphonic acid cation exchange resin (Amberlyst-15) of different particle sizes, with alkali salts (lithium chloride, sodium chloride, potassium chloride, lithium sulphate, sodium sulphate and potassium sulphate)

have been studied with a wiew to compare with those with alkaloid sulphates.

The results indicate that selectivity coefficients for Li-H, Na-H and K-H exchange are less in the chloride solutions than in the sulphate solutions. As the mole fraction of the counter ion in the resin phase, $\bar{X}_M$ increases, the selectivity coefficient decreases. The order of the selectivity is mostly Li < H < Na < K in the chloride solution, while it is H < Li < Na < K in the sulphate solution. The variation of the particle size does not have any significant effect on the exchange equilibria within the range studied. It appears that the resin is of structurally different type than the conventional resins.

6. Equilibria with cinchona alkaloid sulphates :

The exchange equilibria with quinidine sulphate and cinchonine sulphate in dilute aqueous solution with various cation exchange resins have been studied. The variable studied are the relative degree of crosslinking of the resin, the particle size of the resin and the ratio of resin concentration to alkaloid sulphates concentration. The behaviour with alkaloid sulphates is of different type as compared to that of simple alkali cations. For resins of higher degree of crosslinking, the same fraction of resin capacity is exchanged irrespective of the ratio of resin concentration to the alkaloid sulphate concentration. This value varies with the relative degree of crosslinking of the resin. For low crosslinked resins, the effective exchange

capacity is, to a small extent dependent on the ratio of the resin concentration to alkaloid sulphate concentration.

7. Exchange of alkaloid sulphates in presence of different concentrations of sulphuric acid :

Exchange equilibria of quinine, quinidine, cinchonine and cinchonidine sulphates in the presence of added sulphuric acid of different concentrations has been studied with Amberlite IR-200, Amberlyst-15, Dowex 50-X4 and Dowex 50-X8 resins.

8. Uptake of cinchona alkaloid base in 20 % ethanol and ethanol by Dowex 50-X8 and Amberlite IR-200 resins :

A study has been made of the effect of concentration and volume on the uptake of quinine and cinchonine bases from alcoholic solution with Amberlite IR-200 and Dowex 50-X8. The uptake of quinine from aqueous alcoholic solution with Amberlite IR-200 has also been studied.

9. Column study of cinchonine sulphate in aqueous solution :

A preliminary study of the column behaviour of cinchonine sulphate in aqueous solution with different resins has been carried out. Elution has been studied with aqueous sulphuric acid of different concentrations and then by liberation of the cinchonine base with caustic soda solution and elution with distilled ethanol. The results obtained are considered for their value towards practical application.

References :

1. Ultraviolet absorption spectra of coumarins ; Shah,R.S. and Bafna,S.L., Indian.J.Chem., 1, 400 (1963).

2. Molecular sorption on ion-exchange resins : sorption of coumarins ; Shah,R.S. and Bafna,S.L., J.Indian Chem. Soc., 42, 179 (1965).

3. Separation of coumarins ; Shah,R.S. and Bafna,S.L., Nature, 208, 76 (1965).

4. Ultraviolet absorption spectra of cinchona alkaloid sulphates ; Kanhere,S.S., Shah,R.S. and Bafna,S.L., Ind.J.Chem., 3, 251 (1965).

5. Selectivity coefficients of sulphonic acid cation exchange resins in sulphate solution ; Kanhere, S.S., Patel,D.J., Shah, R.S., Bhatt, R.A. and Bafna, S.L., J.Ind.Chem.Soc., 42, 589 (1965) ; Errata (Nov. 1965).