6. <u>COLUMN STUDIES WITH QUININE SULFATE</u>

6. COLUMN STUDIES WITH QUININE SULFATE :

6.1

Introduction :

Earlier (119) column studies with resin Amberlite-200 had been carried out, this study includes column runs with resin X4. The variables studied include (a) flow rate, (b) particle size of the resin, (c) ionic form of the resin and (d) solvent medium.

6.2

Experimental :

Resin X4 of 20/50 and 100/200 mesh was used. Known weights of the airdry resins were taken, slurried with distilled water and transferred to the columns fitted with zero porosity sintered glass discs. The columns were backwashed with distilled water, allowed to settle under gravity and the column data were determined.

6.3

Nomenclature :

W_1 = the amount in milliequivalents of the alkaloid eluted per sample.

TW_x = the total amount in milliequivalents of the alkaloid exchanged.

TW_1 = the total amount in milliequivalents of the alkaloid eluted.

6.4

Results :

Runs were carried out to study exchange with quinine sulfate and elution with N/10 ammonical ethyl alcohol. Five runs were carried out.

A column of resin Dowex 50W X4 of 20/50 mesh in hydrogen form was used. The column data were :
bed length = 32 cms ; bed volume = 14.0 cc. ; capacity of the resin in the column = 16.3 meq. ; P_R = 49.4 (116).
Hence effective capacity for quinine sulfate = 8.05 meq.

Run 1 :

Four litres of aqueous quinine sulfate solution (concentration = 2 meq.per litre) were passed through the column at the rate of 20 cc. per minute. The effluent was collected in one sample and the milliequivalents of quinine sulfate exchanged (TW_x) were calculated by measuring ultraviolet absorption at invarient wavelength (114).

Then the column was washed with distilled water and eluted with 0.1 N ammonical ethyl alcohol at a flow rate of 20 cc. per minute. Sixteen effluent samples were collected, each of 100 cc. and the milliequivalents of quinine eluted (W_1) for each sample were calculated by measuring ultraviolet absorption. After sixteenth sample run was discontinued.

Then the resin in the column was replaced by an equivalent amount of resin from stock, backwashed and allowed to settle under gravity ; then the column was ready for the next run.

Run 2 :

This was the repetition of run 1 except that four litres of aqueous quinine sulfate solution (concentration = 2 meq. per litre) were passed at a flow rate of 4 cc. per minute and elution was also carried out at a flow rate of 4 cc. per minute.

Run 3 :

A column of resin Dowex 50W-X4 of 100/200 mesh in hydrogen form was used. The column data were : bed length = 28 cms ; bed volume = 12 cc. ; capacity of the resin in the column = 16.3 meq. ; P_R = 54.8 (119). Hence effective capacity for quinine sulfate = 8.93 meq.

Run 3 was the repetition of run 2 except that resin used in this case was X4 of 100/200 mesh instead of 20/50 mesh.

Run 4 :

This was the repetition of run 3 except that the resin was converted in ammonium form before starting the run.

Run 5 :

This was the repetition of run 4 except that the influent quinine sulfate solution was in N/100 sulfuric acid solution instead of water.

Table 6.1

Data for the run 1

Resin	=	Dowex 50W X4 (20/50)
Ionic form	=	Hydrogen form
Rate for exchange and elution	=	20 cc./minute
TW_x	=	4.90

Sample No.	500 W_1
1	126.0
2	55.5
3	40.0
4	28.0
5	25.5
6	24.5
7	21.0
8.	20.2
9	19.2
10	19.0
11	18.6
12	18.0
13	17.6
14	17.2
15	15.4
16	14.3

TW_1 = 0.96

Table 6.2

Data for the run 2

Resin	=	Dowex 50W X4 (20/50)
Ionic form	=	Hydrogen form
Rate for exchange and elution	=	4 cc./minute
TW_x	=	5.55

Sample No.	500 W_1
1	1265.0
2	440.6
3	159.1
4	135.0
5	104.0
6	104.0
7	86.0
8	70.5
9	19.0
10	14.7
11.	14.1
12	14.0
13	13.1
14	12.0

TW_1 = 4.90

Table 6.3

Data for the run 3

Resin	=	Dowex 50W X4 (100/200)
Ionic form	=	Hydrogen form
Rate for exchange and elution	=	4 cc./minute
TW_x	=	7.25

Sample No.	500 W_1
1	1455.2
2	1050.0
3	388.2
4	218.6
5	148.2
6	100.0
7	68.4
8	56.5
9	50.2
10	35.5
11	21.2
12	19.2
13	7.2
14	4.0
15	2.6

TW_1 = 7.25

Table 6.4

Data for the run 4

Resin	=	Dowex 50W X4 (100/200)
Ionic form	=	Ammonium form
Rate for exchange and elution	=	4 cc./minute
TW_x	=	7.29

Sample No.	=	500 W_1
1		1705.0
2		802.0
3		327.6
4		326.0
5		191.0
6		139.2
7		49.6
8		32.7
9		23.4
10		16.2
11		13.8
12		10.2
13		4.6
14		3.7

TW_1 = 7.29

Table 6.5

Data for the run 5

Resin	=	Dowex 50W X4 (100/200)
Ionic form	=	Ammonium form
Rate for exchange and elution	=	4 cc./minute
TW_x	=	7.29

Sample No.	500 W_1
1	1520.0
2	1145.0
3	390.5
4	220.2
5	168.5
6	125.8
7	24.8
8	13.8
9	10.0
10	9.1
11	8.6
12	6.2
13	3.2

TW_1 = 7.29

Table 6.6

Total amount of quinine sulfate exchanged (TW_x) and eluted (TW_1)

Run No.	TW_x	TW_1
1	4.90	0.96
2	5.55	4.90
3	7.25	7.25
4	7.29	7.29
5	7.29	7.29

6.5

Discussion :

Runs 1 and 2 indicate that with resin X4 of 20/50 mesh, the decrease in flow rate from 20 cc. to 4 cc. per minute significantly increases the exchange of quinine as indicated by the value of TW_x . However, even at the flow rate of 4 cc. per minute, the value for TW_x is less than that expected from the effective capacity of the resin in the column.

The elution with N/10 ammonical ethyl alcohol is poor as indicated by the value of TW_1 . The elution is relatively better with low flow rate (Table 6.6).

The data of run 3 indicate that resin of smaller mesh size (100/200 mesh) gives the relatively higher value of TW_x . Elution with N/10 ammonical ethyl alcohol at a rate of 4 cc. per minute gives almost practically complete elution.

Run 4 indicates that conversion of resin in NH_4^+ form gives almost the same values of TW_x and TW_1 .

Run 5 indicates that the results are almost same when the solvent medium for quinine sulfate solution is water or N/100 sulfuric acid.

Results of this study, together with those of earlier work (119) with resin IR-200, indicate that the resin of relatively low crosslinking such as X4 of small particle size or of expanded structure such as IR-200 in ammonium form at low flow rate may be considered for more detail work for recovery of cinchona alkaloids with aqueous sulfuric acid as solvent for exchange run and ammonical ethyl alcohol as solvent for elution run.