7. COLUMN STUDIES WITH ALKALI AND CINCHONA ALKALOID SULPHATES :

7.1 Column studies with sodium sulphate and quinine sulphate.

7.1.a. Introduction :

In earlier sections, the study of exchange equilibria and exchange rates of cinchona alkaloids with sulphonic acid cation exchange resins was described. In this section, a preliminary study of relative column behaviour of sodium sulphate and quinine sulphate is described. The variables studied are (a) the degree of cross-linking, (b) the particle size and (c) the structure of the resin. Elution has been studied with N/10 sulphuric acid, N sulphuric acid and ethanol, (after the base was liberated in the resin particle with sodium hydroxide).

7.1.b. Experimental :

Resins : The resins were from the same samples which have been used earlier.

Chemicals : Recrystallised quinine sulphate used was from the stock used earlier. Sodium sulphate (A.R.) , caustic soda (A.R.), sulphuric acid (A.R.) and ethanol (distilled), were used.

Solutions : Preparation of aqueous quinine sulphate solution and its estimation were carried out in the same way as described earlier. Stock solution of aqueous sodium sulphate of strength 0.01 N was prepared in distilled water.

Procedure for column studies :

Preparation of the column : A known weight of the air-dry resin was taken and slurried with distilled water. This slurry was transferred to a column (length $\sim$ 125 cms. and diameter $\sim$ 1 cm. ; fitted with a zero porosity disc near the bottom). The column was back-washed with distilled water and then the resin was allowed to settle down slowly under gravity and bed-volume was estimated. The weight of the resin taken was so adjusted as to give a bed-length of **18 cms.** for each resin studied.

Run (a). Exchange with sodium sulphate solution :

The sodium sulphate solution was then run in the column prepared as above, and the effluent samples was collected (each of 250 cc.) with a flow rate of 10 cc./ minute. The effluent sample was estimated for sulphuric acid by titration against standard sodium hydroxide solution. From the titre values, the % exchange per sample was calculated, from % exchange = Co.100/Ci , where Co represents the concentration of liberated sulphuric acid and Ci represents the concentration of sodium sulphate in influent solution.

Run (b). Exchange with quinine sulphate solution :

The resin in the above column was regenerated by passing slowly sufficient excess of 1 N hydrochloric acid. The resin was then washed free of acid with distilled water, back-washed and allowed to settle down under gravity.

Quinine sulphate solution (concentration = 2 meq. / litre) was then passed through the column and the effluent was collected in samples of suitable volumes at a flow rate of 10 cc. / minute.

The concentration of quinine sulphate, in each sample was estimated by measuring the optical density at 296.5 mμ with or without suitable dilution with distilled water. Knowing the optical density of the influent quinine sulphate solution at the same wave-length and with the same extent of dilution, the % exchange of quinine sulphate per sample was calculated from % exchange = (Di - Do).100/Di , where Do represents the observed optical density at 296.5 mμ after suitable dilution with distilled water, and Di represents the optical density of the influent quinine sulphate solution at the same wave-length with the same extent of dilution. After the run was over, the column was washed with about five bed-volumes quantity of distilled water.

Run (c). Elution of the column :

(i) Elution with N/10 sulphuric acid : N/10 sulphuric acid was passed in the column and the effluent was collected in samples (each of 250 cc.) at a flow rate of 10 cc./ minute. The concentration of quinine sulphate in each sample of the effluent was estimated by measuring the optical density, at 296.5 mμ of each sample with or without suitable dilution with distilled water. Knowing the optical density of the influent quinine sulphate solution of run (b), at the same wave-length and with the same dilution, the % elution of quinine sulphate per sample was calculated from , Do.100/Di. as given above. Flow of N/10 sulphuric acid was discontinued after some samples had been collected.

(ii) Elution with 1 N sulphuric acid : Then 1 N sulphuric acid was passed in the above column and the effluent was again collected in samples (each of 250 cc.) at the same flow rate. Several such samples were collected and the % elution for each sample was calculated as described above.

(iii) Elution with ethanol : The flow of 1 N sulphuric acid was then stopped, acid washed with water, and an excess of 1 N sodium hydroxide solution was passed through the column at a slow rate, to liberate the quinine in the resin phase. Ethanol was then passed in the column to elute the liberated quinine. The effluent was collected at a flow rate of 1 cc./minute, (1) in samples (each of 10 cc.) for resins X 12 and X 16 and (2) in one lot in other cases. The optical density of the effluent was noted at 296.5 mμ after suitable dilution. Knowing the optical density of the quinine solution in alcohol (concentration, 2 meq./litre) at the same wave-length and with the same dilution, the % elution of quinine, in each sample for (1), and the total quinine present in a single lot of the effluent for (2), was calculated. The run was then discontinued.

The column was then emptied out and a column of the next resin was set up. The run was then repeated in the same way as above. In this way, runs were carried out with resins X 4 , X 8 , X 12 ; X 16 and X 20 and resins IR-200 and IR-120 of different particle diameters. The results are given in the following tables.

7.1.c. RESULTS :

Tables (7.01.a. to 7.05.a.) give the data for the exchange with sodium sulphate solution in columns of resins X 4 to X 20.

Tables (7.06.a. to 7.09.a.) give the data for the exchange with sodium sulphate solution in columns of resin IR-200 of different particle diameters.

Tables (7.10.a. to 7.12.a.) give the data for the exchange with sodium sulphate solution in columns of resins IR-120 of different particle diameter.

Tables (7.01.b. to 7.05.b.) give the data for the exchange with quinine sulphate solution in columns of resins X 4 to X 20.

Tables (7.06.b. to 7.09.b.) give the data for the exchange with quinine sulphate solution in columns of resin IR-200 of different particle daimeter.

Tables (7.10.b. to 7.12.b.) give the data for the exchange with quinine sulphate solution in columns of resin IR-120 of different particle diameter.

Tables (7.01.c.to 7.05.c.)give the data for elution from the columns of resins X 4 to X 20.

Tables (7.06.c. to 7.09.c.) give the data for elution from the columns of resin IR-200 of different particle diameter.

Tables (7.10.c. to 7.12.c.) give the data for elution from the columns of resins IR-120 of different particle diameter.

Tables (7.13.a. , 7.13.b. and 7.13.c.) give the break-through capacity per meq. for resins X 4 to X 20 and resins IR-200 and IR-120 (of different particle diameter).

Figures (7.01 to 7.05) give the plot of % exchange with sodium sulphate solution and quinine sulphate solution against the sample number for resins X 4 to X 20 .

Figure (7.06) gives the plot of % exchange with sodium sulphate solution and quinine sulphate solution against sample number for resin IR-200 of different particle diameter.

Figure (7.07) gives the plot of % exchange with sodium sulphate solution and quinine sulphate solution against sample number for resin IR-120 of different particle diameter.

Figure (7.08) gives the plot of % elution of quinine sulphate against sample number with N/10 and 1 N sulphuric acid as eluant from columns of resins X 4, X 8 and X 12 .

Figure (7.09) gives the plot of % elution of quinine sulphate against sample number with N/10 and 1 N sulphuric acid as eluant from a column of resin IR-200 of different particle diameter.

Figure (7.10) gives the plot of % elution of quinine sulphate against sample number with 1 N sulphuric acid as eluant from the columns of resin IR-120 of different particle diameter ; and the plot of % elution of quinine against sample number with ethanol as eluant for resin X 16 .

Figure (7.11) gives the plot of break-through capacity per meq. of total capacity of resins X 4 to X 20 ; IR-200 and IR-120, of different particle diameter.

(Break-through capacity was calculated at 99 % exchange for each run.)

Figure 7.01

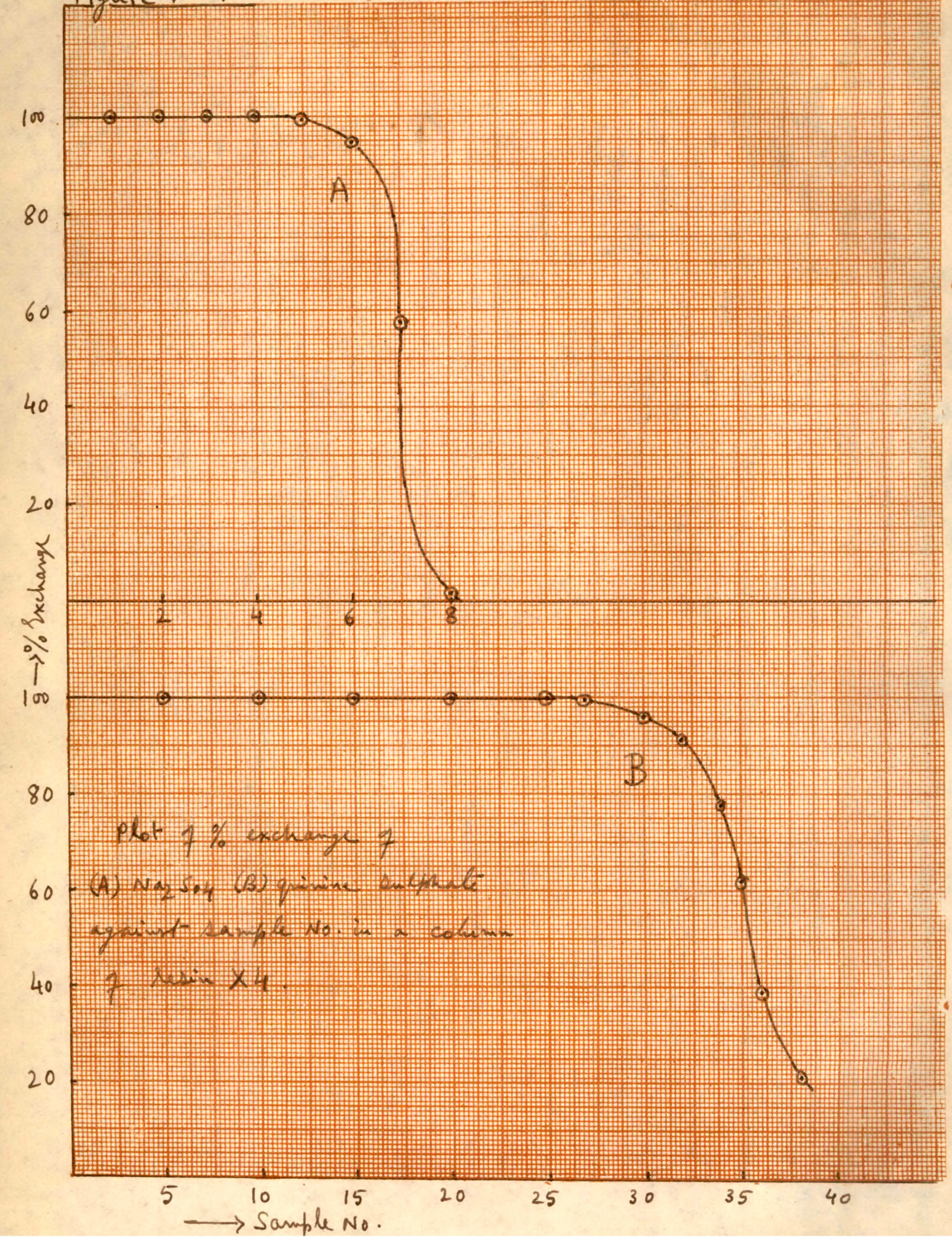

100
80
60
40
20
A
2
4
6
8
100
80
60
40
20
B
→ % Exchange
Plot of % exchange of
(A) Na_2SO_4 (B) quinine sulphate
against sample No. in a column
of resin X4.
5
10
15
20
25
30
35
40
→ Sample No.

Table 7.01.a.

Exchange with sodium sulphate solution in a column of resin X 4.

Column capacity = 16.3 meq.

Sample Number	Sample volume in cc.	% exchange	Sample Number	Sample volume in cc.	% exchange
1	250	100.0	5	250	99.06
2	250	100.0	6	250	94.39
3	250	100.0	7	250	57.20
4	250	100.0	8	250	1.40

Table 7.01.b.

Exchange with quinine sulphate solution, in the above column of resin X 4, after regeneration.

1	100	100.0	25	100	99.61
3	100	100.0	27	100	99.61
5	100	100.0	28	100	98.09
7	100	100.0	29	100	97.14
9	100	100.0	30	100	96.38
11	100	100.0	32	100	91.62
13	100	99.81	34	100	77.38
15	100	99.81	35	100	61.79
17	100	99.81	36	100	39.16
23	100	99.81	38	100	21.10

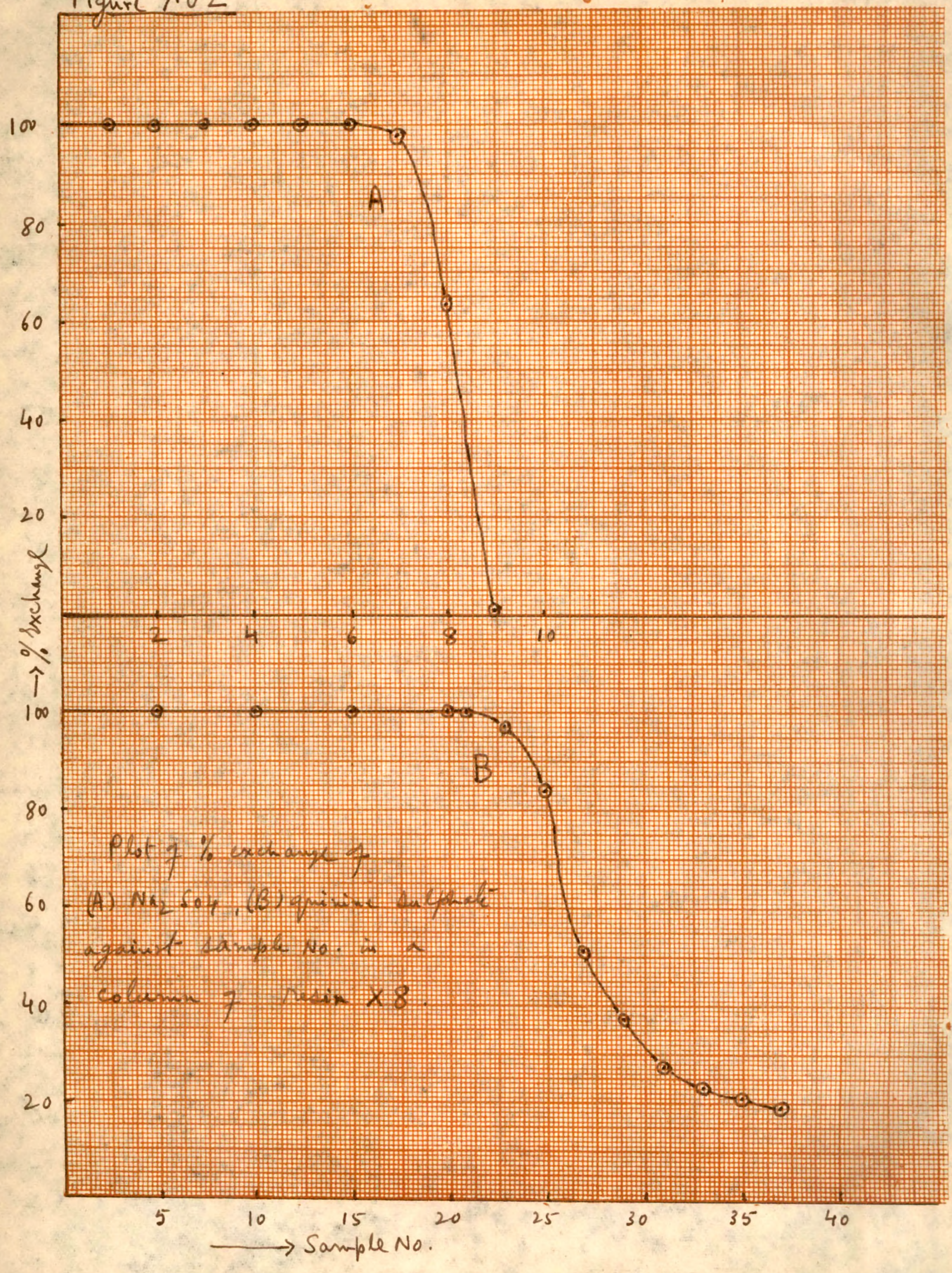
Table 7.01.c.

Elution of quinine sulphate / quinine from the above column of resin X 4.

Eluant	Sample No.	Sample volume in cc.	% elution
N/10	1	250	5.89
H_2SO_4	2	250	7.52
(flow rate	3	250	7.22
10 cc./min.)	4	250	6.81
	5	250	6.42
1 N			
H_2SO_4	1	250	130.60
(flow rate	2	250	88.12
10 cc./min.)	3	250	74.15
	4	250	64.45
	5	250	58.65
	6	250	54.67
	7	250	50.76
	8	250	43.06
	9	250	40.12

Ethanol (One lot of 100 cc. at 1 cc./min.)
2.38 meq. of quinine was found in the effluent.
After this the run was discontinued.

Figure 7.02



Plot of % exchange of (A) Na_2SO_4, (B) quinine sulphate against sample No. in a column of resin X8.

Table 7.02.a.

Exchange with sodium sulphate solution in a column of resin X 8.

Column capacity = 19.04 meq.

Sample Number	Sample volume in cc.	% exchange	Sample Number	Sample volume in cc.	% exchange
1	250	100.0	6	250	100.0
2	250	100.0	7	250	97.68
3	250	100.0	8	250	62.79
4	250	100.0	9	250	0.93
5	250	100.0	-	-	-

Table 7.02.b.

Exchange with quinine sulphate solution in the above column of resin X 8, after regeneration.

1	100	100.0	27	100	51.14
10	100	100.0	29	100	36.88
19	100	100.0	31	100	27.18
21	100	99.81	33	100	23.38
22	100	97.90	35	100	20.91
23	100	97.34	37	100	19.20
25	100	83.47	-	-	-

Table 7.02.c.

Elution of quinine sulphate / quinine from the above column of resin X 8 .

Eluant	Sample No.	Sample volume in cc.	% elution
N/10	1	250	0.52
H_2SO_4	2	250	0.44
(flow rate 10 cc./min.)	3	250	0.44
1 N	1	250	43.80
H_2SO_4	2	250	33.05
(flow rate	3	250	27.61
10 cc./min.)	4	250	24.57
Ethanol	(One lot of 100 cc. at 1 cc./ min.)		

1.88 meq. of quinine was found in 100 cc. of the effluent.

After this the run was discontinued.

Figure 7.03

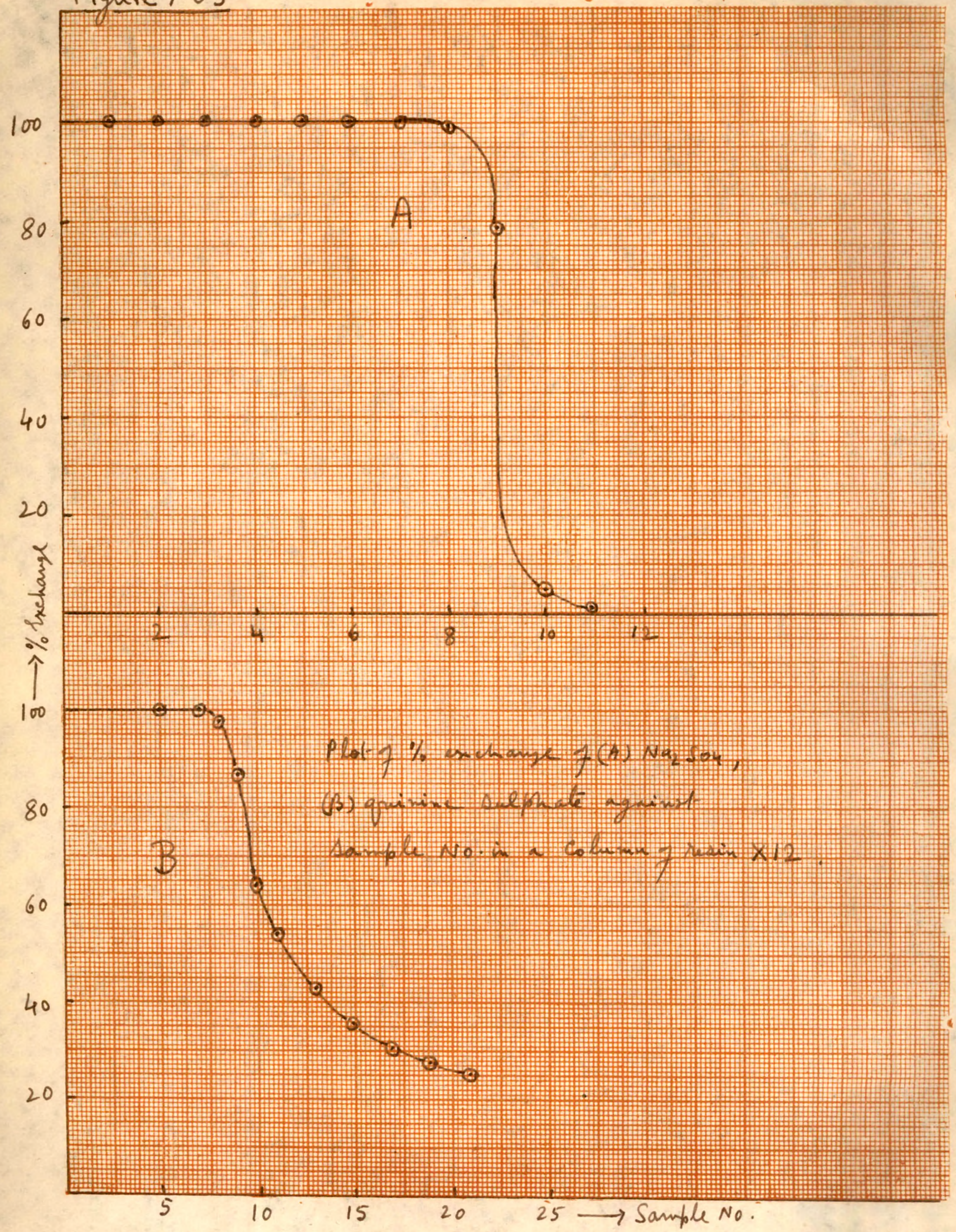

100
80
60
40
20
A
2
4
6
8
10
12
→ % Exchange
100
80
60
40
20
B
5
10
15
20
25 → Sample No.
Plot of % exchange of (A) Na2SO4,
(B) quinine sulphate against
sample No. in a column of resin X12.

Table 7.03.a.

Exchange with sodium sulphate solution in a column of resin X 12.

Column capacity = 22.04 meq.

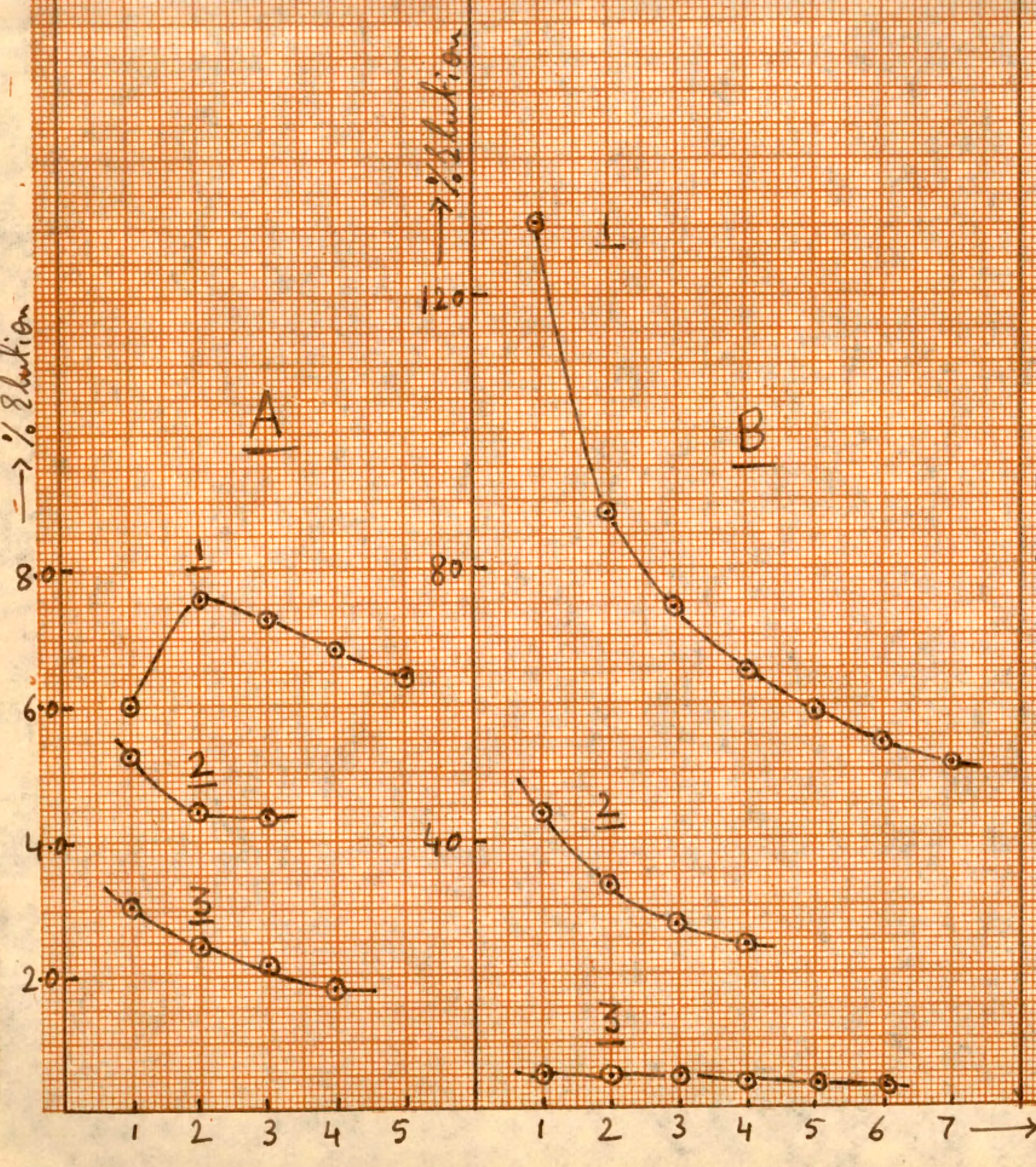
Sample Number	Sample volume in cc.	% exchange	Sample Number	Sample volume in cc.	% exchange
1	250	100.0	6	250	100.0
2	250	100.0	7	250	100.0
3	250	100.0	8	250	99.06
4	250	100.0	9	250	78.14
5	250	100.0	10	250	3.72
-	-	-	11	250	0.46

Table 7.03.b.

Exchange with quinine sulphate solution in the above column of resin X 12 after regeneration.

1	100	100.0	11	100	53.99
3	100	100.0	13	100	43.15
5	100	99.81	15	100	35.93
7	100	99.81	17	100	30.98
8	100	97.52	19	100	28.52
9	100	86.50	21	100	25.48
10	100	64.25	-	-	-

Figure 7.08



Plot of % elution of quinine sulphate against sample No. with (A) N/10 H_2SO_4 ; (B) 1N H_2SO_4, from a Column of

1 resin × 4

2 resin × 8

3 resin × 12

Table 7.03.c.

Elution of quinine sulphate/quinine from the above column of resin X 12 .

Eluant	Sample No.	Sample volume in cc.	% elution
N/10	1	250	0.30
H_2SO_4	2	250	0.24
(flow rate	3	250	0.22
10 cc./min.)	4	250	0.18
1 N H_2SO_4	1	250	4.49
(flow rate	2	250	4.80
10 cc./min.)	3	250	4.49
	4	250	4.15
	5	250	3.90
	6	250	3.68
Ethanol	1	10	10.66
(flow rate	2	10	3.33
1 cc./min.)	3	10	3.23
	4	10	2.17
	5	10	1.15

After this the run was discontinued.

Figure 7.04

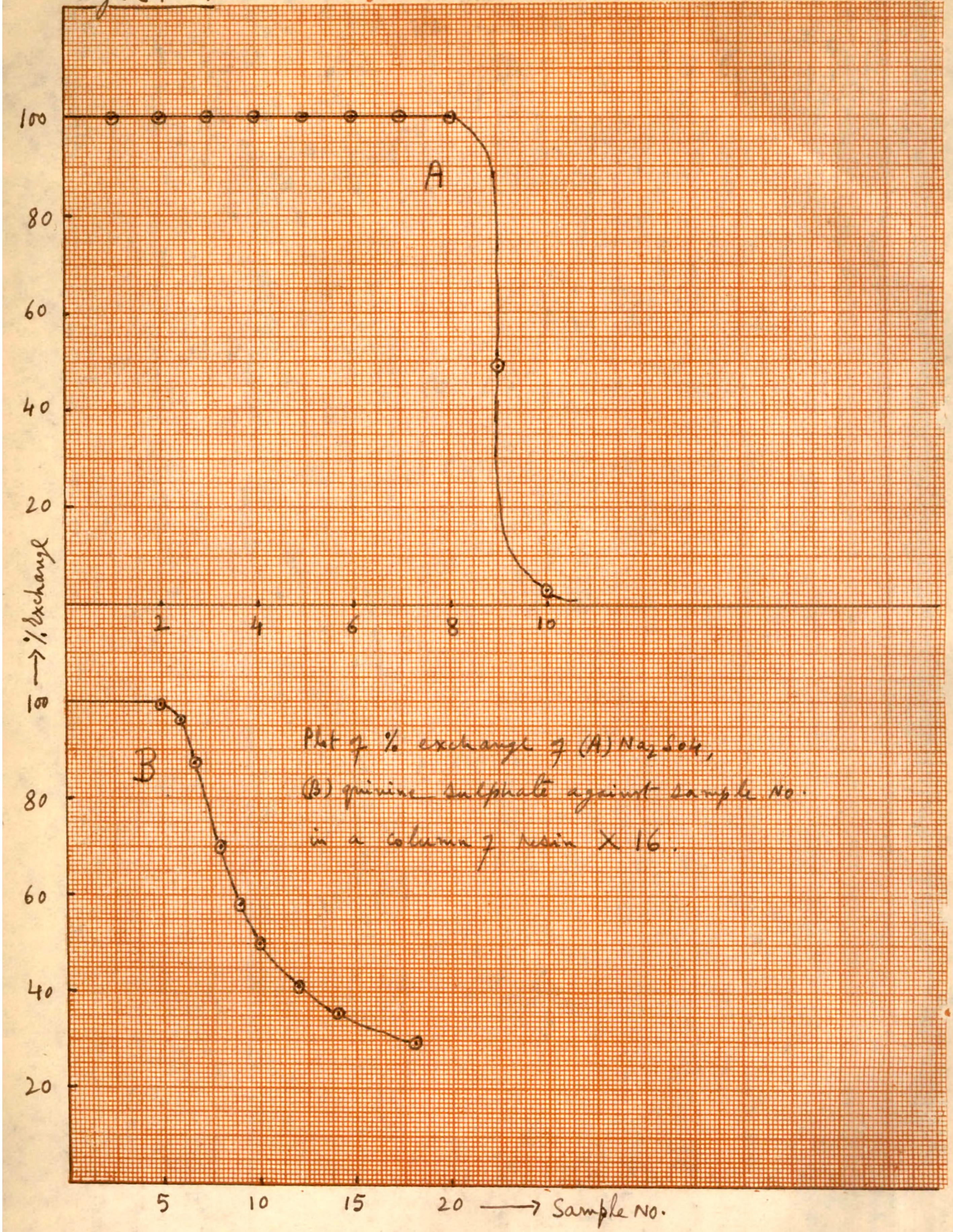

100
80
60
40
20
A
2
4
6
8
10
% Exchange
100
80
60
40
20
B
5
10
15
20
Sample No.
Plot of % exchange of (A) Na2SO4,
(B) quinine sulphate against sample No.
in a column of resin X 16.

Table 7.04.a.

Exchange with sodium sulphate solution in a column of resin X 16.

Column capacity = 21.3 meq.

Sample Number	Sample volume in cc.	% exchange	Sample Number	Sample volume in cc.	% exchange
1	250	100.0	6	250	100.0
2	250	100.0	7	250	100.0
3	250	100.0	8	250	100.0
4	250	100.0	9	250	48.84
5	250	100.0	10	250	2.79

Table 7.04.b.

Exchange with quinine sulphate solution in the above column of resin X 16, after regeneration.

1	50	100.0	8	50	69.95
3	50	100.0	9	50	57.41
4	50	99.81	10	50	49.43
5	50	99.61	12	50	40.68
6	50	96.20	14	50	34.97
7	50	87.26	18	50	28.89

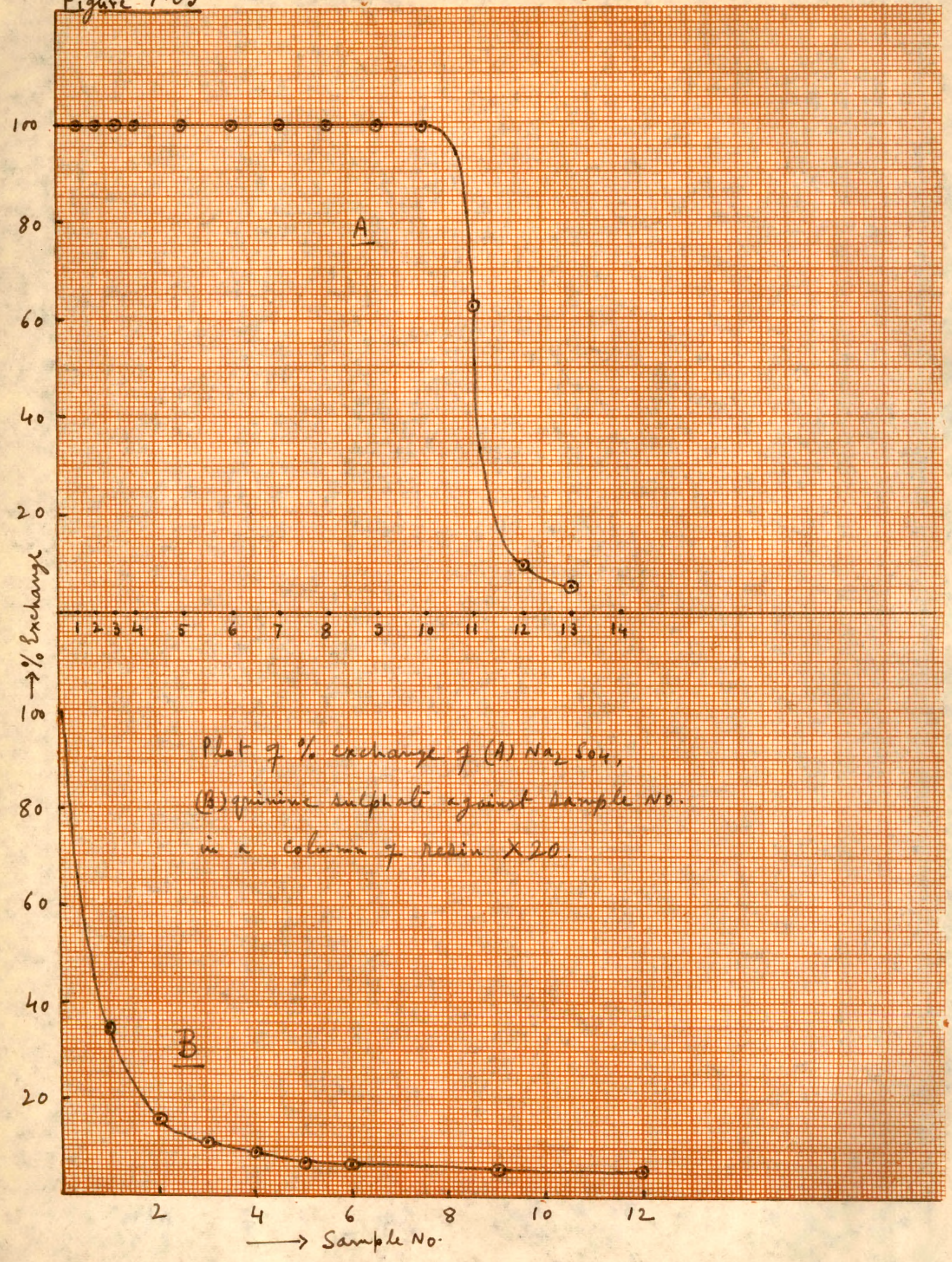
Table 7.04.c.

Elution of quinine from the above column of resin X 16.

Eluant	Sample No.	Sample volume in cc.	% elution
Ethanol	1	10	7.54
(flow rate	2	10	4.93
1 cc./min.)	3	10	3.20
	4	10	2.40
	5	10	1.90

After this the run was discontinued.

Figure 7.05



Plot of % exchange of (A) Na_2SO_4, (B) quinine sulphate against sample No. in a column of resin X20.

Table 7.05.a.

Exchange with sodium sulphate solution in a column of resin X 20 .

Column capacity = 21.0 meq.

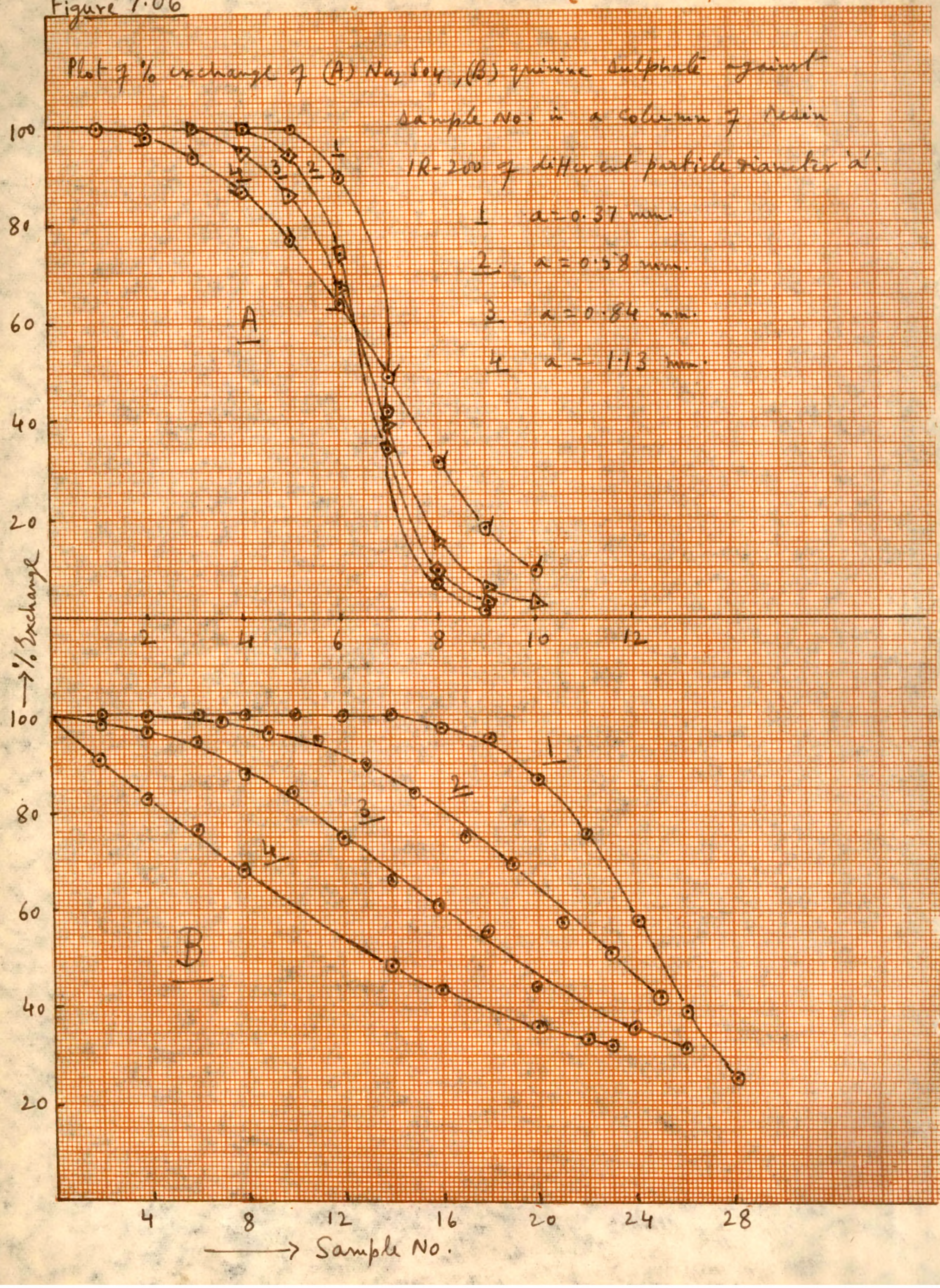
Sample Number	Sample volume in cc.	% exchange	Sample Number	Sample volume in cc.	% exchange
1	100	100.0	8	250	100.0
2	100	100.0	9	250	100.0
3	100	100.0	10	250	100.0
4	100	100.0	11	250	63.25
5	250	100.0	12	250	9.77
6	250	100.0	13	250	5.58
7	250	100.0	-	-	-

Table 7.05.b.

Exchange with quinine sulphate solution in the above column of resin X 20, after regeneration.

1	50	35.37	5	50	7.59
2	50	15.74	6	50	6.85
3	50	11.48	9	50	6.11
4	50	9.44	12	50	5.74

Figure 7.06



Plot of % exchange of (A) Na_2SO_4, (B) quinine sulphate against sample No. in a column of resin IR-200 of different particle diameter 'a'.
1 a = 0.37 mm.
2 a = 0.58 mm.
3 a = 0.84 mm.
4 a = 1.13 mm.
A
B
1
2
3
4
100
80
60
40
20
2
4
6
8
10
12
16
20
24
28
% Exchange
Sample No.

Table 7.06.a.

Exchange with sodium sulphate solution in a column of resin IR-200 (a = 1.13 mm.)

Column capacity = 15.67 meq.

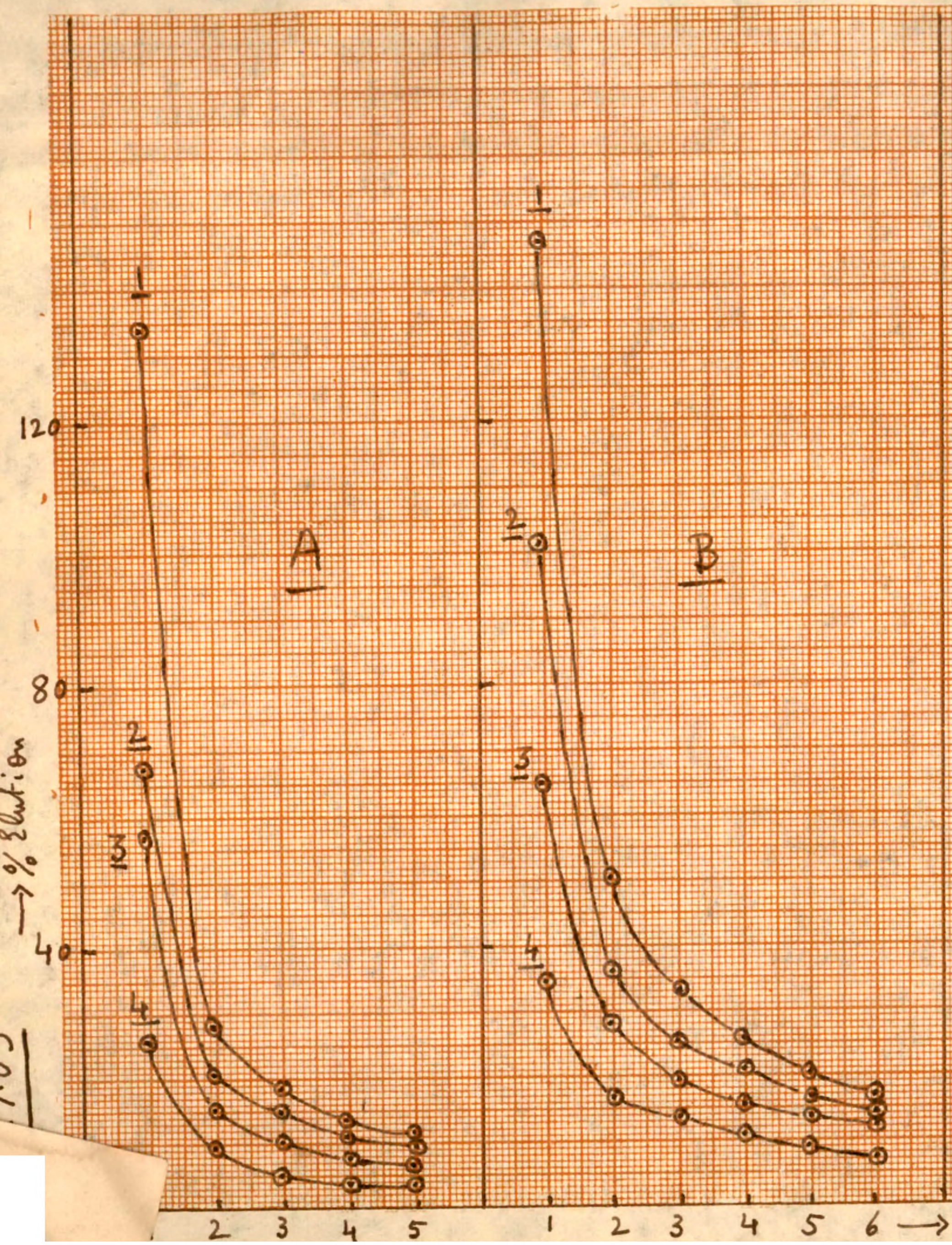
Sample Number	Sample volume in cc.	% exchange	Sample Number	Sample volume in cc.	% exchange
1	250	100.0	6	250	63.95
2	250	97.86	7	250	48.45
3	250	93.99	8	250	31.00
4	250	87.68	9	250	17.44
5	250	77.03	10	250	9.69

Table 7.06.b.

Exchange with quinine sulphate solution in the above column of resin IR-200 after regeneration.

2	100	91.68	16	100	45.16
4	100	83.35	18	100	43.99
6	100	77.34	20	100	36.64
8	100	68.80	22	100	34.31
10	100	64.92	24	100	33.14
12	100	50.58	26	100	33.14
14	100	49.22	-	-	-

7.09



Plot of % elution of quinine sulphate against sample No. with (A) N/10 H_2SO_4, (B) 1N H_2SO_4, from a column of resin IR-200 of different particle diameter 'a',

1 a = 0.37 mm.

2 a = 0.58 mm.

3 a = 0.84 mm.

4 a = 1.13 mm.

Table 7.06.c.

Elution of quinine sulphate/quinine from the above column of resin IR-200.

Eluant	Sample No.	Sample volume in cc.	% elution
N/10 H_2SO_4 (flow rate 10 cc./min.)	1	250	26.60
	2	250	8.97
	3	250	5.35
	4	250	4.34
	5	250	3.78
1 N H_2SO_4 (flow rate 10 cc./min.)	1	250	34.17
	2	250	16.27
	3	250	13.77
	4	250	10.51
	5	250	9.26
	6	250	6.92
Ethanol	(one lot of 100 cc. at 1 cc. / min.)		

2.21 meq. of quinine was found in 100 cc. of the effluent.

After this the run was discontinued.

Table. 7. 07. a.

Exchange with sodium sulphate solution in a column of resin IR-200 (a = 0.84 mm.).

Column capacity = 15.32 meq.

Sample Number	Sample volume in cc.	% exchange	Sample Number	Sample volume in cc.	% exchange
1	250	100.0	6	250	67.48
2	250	100.0	7	250	39.32
3	250	100.0	8	250	15.05
4	250	95.15	9	250	6.31
5	250	86.42	10	250	3.40

Table 7. 07. b.

Exchange with quinine sulphate solution in the above column of resin IR-200, after regeneration.

1	100	100.0	14	100	65.85
2	100	98.47	16	100	61.06
4	100	96.77	18	100	56.10
6	100	95.04	20	100	43.89
8	100	88.37	22	100	39.31
10	100	84.55	24	100	36.45
12	100	75.19	26	100	32.25

Table 7.07.c.

Elution of quinine sulphate/ quinine from the above column of resin IR-200.

Eluant	Sample No.	Sample volume in cc.	% elution
N/10 H_2SO_4 (flow rate 10 cc./min.)	1	250	56.18
	2	250	14.96
	3	250	9.92
	4	250	7.69
	5	250	6.26
1 N H_2SO_4 (flow rate 10 cc./min.)	1	250	63.89
	2	250	27.71
	3	250	19.01
	4	250	14.88
	5	250	13.67
	6	250	12.06
Ethanol	(one lot of 100 cc. at 1 cc. /min.). 2.32 meq. of quinine was found in 100 cc. of the effluent. After this the run was discontinued.		

Table 7.08.a.

Exchange with sodium sulphate solution in a column of resin IR-200 (a = 0.58 mm.).

Column capacity = 15.47 meq.

Sample Number	Sample volume in cc.	% exchange	Sample Number	Sample volume in cc.	% exchange
1	250	100.0	6	250	73.72
2	250	100.0	7	250	33.81
3	250	100.0	8	250	8.92
4	250	99.54	9	250	3.29
5	250	93.91	-	-	-

Table 7.08.b.

Exchange with quinine sulphate solution in the above column of resin IR-200, after regeneration.

2	100	100.0	15	100	84.35
4	100	99.63	17	100	75.18
6	100	99.42	19	100	68.71
7	100	99.06	21	100	57.25
9	100	96.38	23	100	50.95
11	100	95.04	25	100	42.37
13	100	90.07	-	-	-

Table 7.08.c.

Elution of quinine sulphate/quinine from the above column of resin IR-200 .

Eluant	Sample No.	Sample volume in cc.	% elution
N/10 H_2SO_4 (flow rate 10 cc./min.)	1	250	67.17
	2	250	19.85
	3	250	14.62
	4	250	11.04
	5	250	8.99
1 N H_2SO_4 (flow rate 10 cc./min.)	1	250	101.50
	2	250	35.65
	3	250	23.82
	4	250	21.07
	5	250	16.18
	6	250	13.67

Ethanol (One lot of 100 cc. at 1 cc./ min.)

2.415 meq. of quinine was found in 100 cc. of the effluent.

After this the run was discontinued.

Table 7.09.a.

Exchange with sodium sulphate solution in a column of resin IR-200 (a = 0.37 mm.).

Column capacity = 16.02 meq.

Sample Number	Sample Volume in cc.	% exchange	Sample Number	Sample volume in cc.	% exchange
1	250	100.0	6	250	89.48
2	250	100.0	7	250	41.63
3	250	100.0	8	250	6.70
4	250	100.0	9	250	2.39
5	250	99.04	-	-	-

Table 7.09.b.

Exchange with quinine sulphate solution in the above column of resin IR-200, after regeneration.

1	100	100.0	18	100	94.97
2	100	99.79	20	100	86.66
6	100	99.79	22	100	75.44
10	100	99.79	24	100	57.63
14	100	99.61	26	100	39.26
16	100	97.09	28	100	25.72

Table. 7. 09. c.

Elution of quinine sulphate/quinine from the above column of resin IR-200 .

Eluant	Sample No.	Sample volume in cc.	% elution
N/10 H_2SO_4 (flow rate 10 cc./min.)	1	250	134.60
	2	250	27.47
	3	250	17.75
	4	250	13.13
	5	250	10.89
1 N H_2SO_4 (flow rate 10 cc./min.)	1	250	146.40
	2	250	49.90
	3	250	32.49
	4	250	25.54
	5	250	19.72
	6	250	16.44

Ethanol (One lot of 100 cc. at 1 cc./min.)

2.28 meq. of quinine was found in 100 cc. of the effluent.

After this the run was discontinued.

Figure 7.07

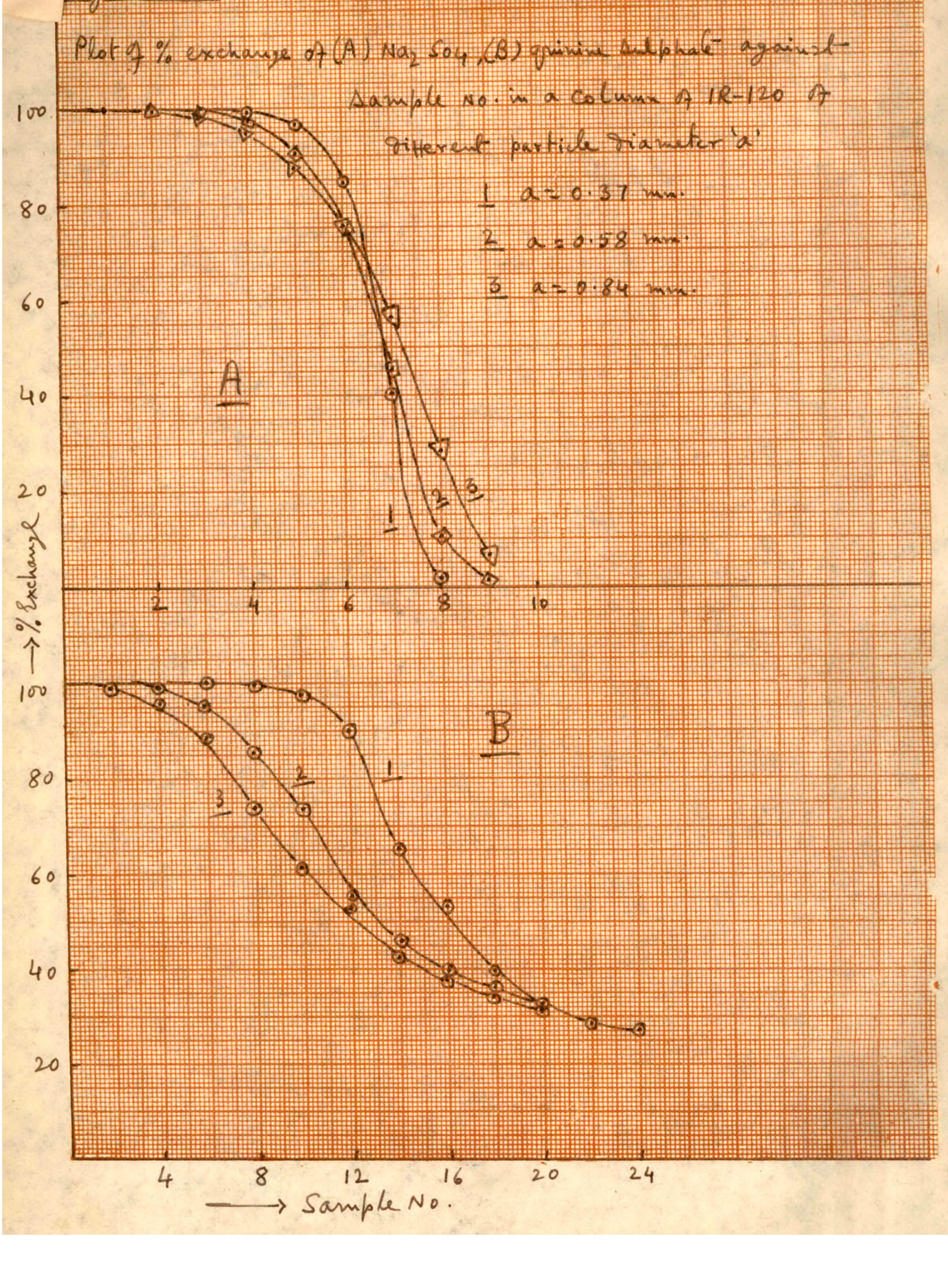

Plot of % exchange of (A) Na_2SO_4, (B) quinine sulphate against Sample No. in a column of IR-120 of different particle diameter 'a'
1 a = 0.37 mm.
2 a = 0.58 mm.
3 a = 0.84 mm.
A
B
1
2
3
100
80
60
40
20
2
4
6
8
10
12
16
20
24
→ % Exchange
→ Sample No.

Table 7.10.a.

Exchange with sodium sulphate solution in a column of resin IR-120 (a = 0.84 mm.).

Column capacity = 16.15 meq.

Sample Number	Sample volume in cc.	% exchange	Sample Number	Sample volume in cc.	% exchange
1	250	100.0	6	250	75.50
2	250	99.02	7	250	55.89
3	250	98.04	8	250	27.46
4	250	94.61	9	250	6.37
5	250	87.27	-	-	-

Table 7.10.b.

Exchange with quinine sulphate solution in the above column of resin IR-120 , after regeneration.

1	100	100.0	12	100	53.46
2	100	98.97	14	100	43.08
4	100	96.34	16	100	38.00
6	100	88.41	18	100	34.95
8	100	74.39	20	100	32.31
10	100	62.38	24	100	26.42

Table 7.11.a.

Exchange with sodium sulphate solution in a column of resin IR-120 (a = 0.58 mm.).

Column capacity = 15.46 meq.

Sample Number	Sample volume in cc.	% exchange	Sample Number	Sample volume in cc.	% exchange
1	250	100.0	6	250	75.01
2	250	100.0	7	250	45.10
3	250	99.49	8	250	9.80
4	250	97.54	9	250	0.98
5	250	90.20	-	-	-

Table 7.11.b.

Exchange with quinine sulphate solution in the above column of resin IR-120 , after regeneration.

1	100	100.0	14	100	45.32
2	100	99.81	16	100	39.63
4	100	98.76	18	100	36.59
6	100	95.10	20	100	33.13
8	100	84.74	22	100	28.25
10	100	73.57	24	100	26.83
12	100	54.88	-	-	-

Figure 7.10

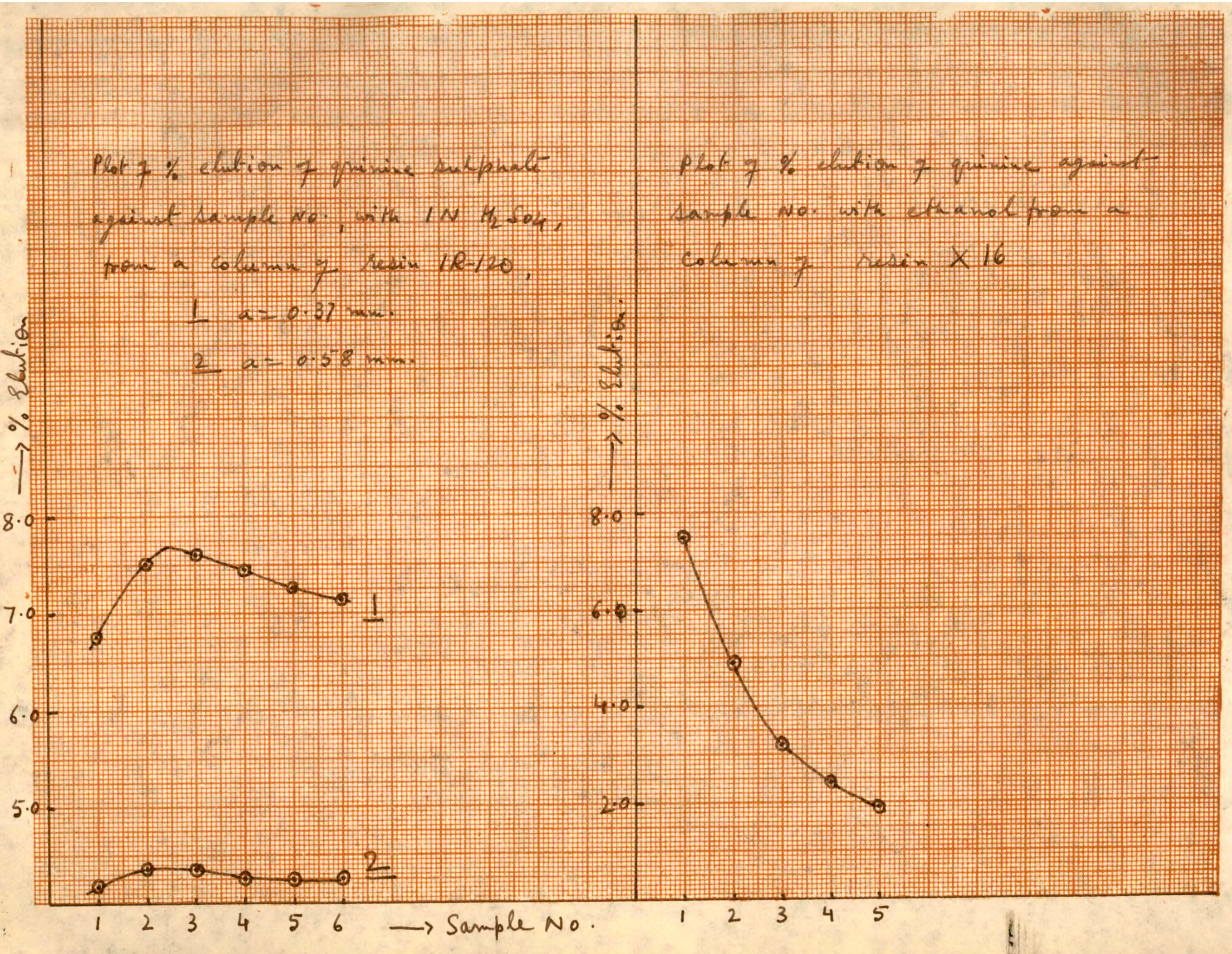

Plot of % elution of quinine sulphate against sample No., with 1N H_2SO_4, from a column of resin IR-120,
1 a = 0.37 mm.
2 a = 0.58 mm.
→ % Elution
8.0
7.0
6.0
5.0
1
2
1 2 3 4 5 6
→ Sample No.
Plot of % elution of quinine against sample No. with ethanol from a column of resin X 16
→ % Elution
8.0
6.0
4.0
2.0
1 2 3 4 5

Table 7.11.c.

Elution of quinine sulphate / quinine from the above column of resin IR-120 .

Eluant	Sample No.	Sample volume in cc.	% elution
1 N H_2SO_4 (flow rate 10 cc. /min.)	1	250	4.13
	2	250	4.34
	3	250	4.34
	4	250	4.23
	5	250	4.23
	6	250	4.23
Ethanol	(One lot of 50 cc. at 1 cc./min.) 0.241 meq. of quinine was found in 50 cc. of the effluent. After this the run was discontinued.		

Table 7.12.a.

Exchange with sodium sulphate solution in a column of resin IR-120 (a = 0.37 mm.).

Column capacity = 15.66 meq.

Sample Number	Sample volume in cc.	% exchange	Sample Number	Sample volume in cc.	% exchange
1	250	100.0	5	250	96.56
2	250	100.0	6	250	84.31
3	250	100.0	7	250	40.20
4	250	99.02	8	250	1.47

Table 7.12.b.

Exchange with quinine sulphate solution in the above column of resin IR-120 , after regeneration.

1	100	100.0	12	100	90.03
2	100	100.0	14	100	64.83
4	100	100.0	16	100	53.24
6	100	99.88	18	100	39.22
8	100	98.97	20	100	32.11
10	100	97.74	22	100	25.20

Table 7.12.c.

Elution of quinine sulphate / quinine from the above column of resin IR-120 .

Eluant	Sample No.	Sample volume in cc.	% elution
1 N	1	250	6.77
H_2SO_4	2	250	7.50
(flow rate	3	250	7.60
10 cc./min.)	4	250	7.46
	5	250	7.26
	6	250	7.13

Ethanol (One lot of 50 cc. at 1 cc. / min.)

0.329 meq. of quinine was found in 50 cc. of the effluent.

After this the run was discontinued.

7.1.d. Discussion :

The conclusions of the tables (7.01.a. to 7.05.a.) are summarised in tables (7.13 a , b , c). It is observed that with sodium sulphate, under the experimental conditions given, the break-through is quite sharp ; as the degree of cross-linking increases from 4 to 20 , the sharpness is decreased some-what, due to reduced rate of diffusion in the resin particle. In all cases, essentially, full column capacity is realised. The break-through capacity per meq. of resin capacity increases when X increases from 4 to 16 and then slightly decreases when X increases from 16 to 20 . This is similar to the observation earlier (Section Two) that the selectivity coefficient increases, reaches a maximum and then decreases as X increases from 4 to 20 .

With quinine sulphate (tables 7.01.b. to 7.05.b.) the break-through is relatively less sharp for X 4 and both the sharpness and the break-through capacity decrease markedly as X increases. With X 20, the break-through capacity is reduced to zero. This marked difference from sodium sulphate is to be attributed to the large molecular size of quinine, and is in accordance with the result (Section Six) that the rate of exchange decreases markedly as X increases. The break-through capacity of quinine sulphate per meq. of total capacity of the resin, as well as, per meq. of effective capacity of the resin (obtained by multiplying the total capacity by P_{Ro} /100) decreases

Figure 7.11

—→ Break-through Capacity.

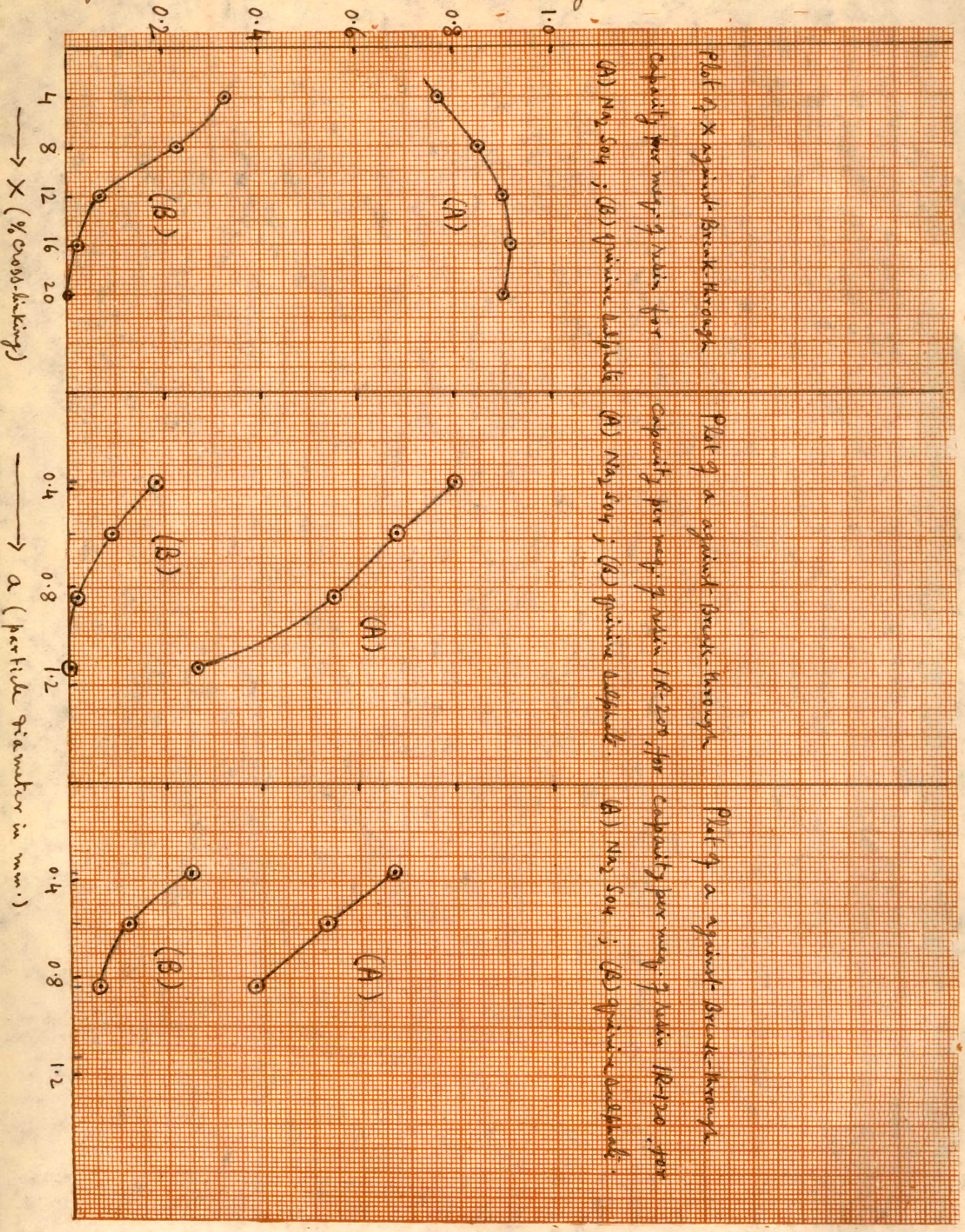


Table 7.13.a.

Summary of column data for tables 7.01 a, b to 7.05 a, b.
The numerator of the fraction in the brackets gives the number of samples and the denominator, the volume of each sample.

Resin	a (mm.)	Column capacity in meq.	P_{Ro}	C_{ef}	exchange with Na_2SO_4 solution in meq.	exchange with quinine sulphate solution in meq.	Break-through capacity in meq., per meq. of total resin capacity with Na_2SO_4 (A)	Quinine sulphate (B)	Break-through capacity in meq., per meq. of effective resin capacity with quinine-sulphate soln.	Y = (A/B)
X 4	0.215	16.3	49.4	8.052	$\frac{16.3}{(8/250)}$	$\frac{6.974}{(38/100)}$	0.7668	0.3311	0.6704	2.316
X 8	0.215	19.04	40.0	7.617	$\frac{19.04}{(9/250)}$	$\frac{5.715}{(37/100)}$	0.8535	0.2258	0.5645	3.780
X12	0.215	22.04	25.5	5.619	$\frac{22.04}{(11/250)}$	$\frac{2.688}{(21/100)}$	0.9069	0.0703	0.2758	12.89
X16	0.215	21.30	18.2	3.877	$\frac{21.30}{(10/250)}$	$\frac{1.143}{(18/50)}$	0.9180	0.0246	0.1352	37.32
X20	0.215	21.00	5.9	1.239	$\frac{21.0}{(4/100 + 9/250)}$	$\frac{0.123}{(12/50)}$	0.9048	zero	-	∞

Table 7.13.b.

Summary of column data for tables 7.06, a.b to 7.12 , a.b

The numerator of the fraction in the brackets gives the number of samples and the denominator, the volume of each sample.

Resin	a (mm.)	Column capacity in meq.	P_{Ro}	C_{ef}	exchange with Na_2SO_4 solution in meq.	exchange with quinine sulphate solution in meq.	Break-through capacity in meq., per meq. of total resin capacity with Na_2SO_4	Break-through capacity in meq., per meq. of total resin capacity with quinine sulphate	Break-through capacity in meq., per meq. of effective resin capacity with quinine-sulphate soln.	Y = (8/9)
1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.
IR-120	0.84	16.15	40.3	6.509	16.15 / (9/250)	2.86 / (24/100)	0.3869	0.02477	0.06146	15.83
	0.58	15.46	40.3	6.230	15.46 / (9/250)	3.138 / (24/100)	0.5335	0.05175	0.1248	10.30
	0.37	15.66	40.3	6.311	15.66 / (8/250)	3.280 / (22/100)	0.6704	0.1028	0.2535	6.522

(Continued on next page)

Table 7.13.b. (continued from the previous page)

1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.
IR-200	1.13	15.67	30.5	4.778	$\frac{15.67}{(10/250)}$	$\frac{2.91}{(26/100)}$	0.2712	zero	-	∞
	0.84	15.32	30.5	4.673	$\frac{15.32}{(10/250)}$	$\frac{3.552}{(26/100)}$	0.5546	0.0235	0.07703	23.60
	0.58	15.47	30.5	4.719	$\frac{15.47}{(9/250)}$	$\frac{4.086}{(25/100)}$	0.6785	0.0905	0.2967	7.498
	0.37	16.02	30.5	4.884	$\frac{16.02}{(9/250)}$	$\frac{4.752}{(28/100)}$	0.7960	0.1823	0.5936	4.366

Table 7.13.c.

Summary of column data for tables 7.01.c. to 7.12.c.

The numerator of the fraction in the brackets gives the number of samples and the denominator, the volume of each sample.

Resin	a (mm.)	Amount in meq., eluted with Eluant: N/10 H_2SO_4	1 N H_2SO_4	Ethanol	Total eluted in meq.	Total uneluted
1.	2.	3.	4.	5.	6.	7.
X 4	0.215	0.1693 / (5/250)	3.023 / (9/250)	2.380 / (1/100)	5.572	1.402
X 8	0.215	0.0070 / (3/250)	0.6451 / (4/250)	1.880 / (1/100)	2.532	3.183
X12	0.215	0.0047 / (4/250)	0.1275 / (6/250)	0.0411 / (5/10)	0.1733	2.515
X16	0.215	-	-	0.04 / (5/10)	0.0400	1.100
IR-120	0.58	______	0.1275 / (6/250)	0.2410 / (1/50)	0.3685	2.769
	0.37	-	0.2186 / (6/250)	0.3290 / (1/50)	0.5476	2.7322

(continued on next page)

Table 7.13.c.

(continued from the previous page.)

1.	2.	3.	4.	5.	6.	7.
IR-200	1.13	0.2452 / (5/250)	0.4545 / (6/250)	2.2100 / (1/100)	2.909	zero
	0.84	0.4750 / (5/250)	0.7561 / (6/250)	2.320 / (1/100)	3.552	zero
	0.58	0.6083 / (5/250)	1.059 / (6/250)	2.415 / (1/100)	4.086	zero
	0.37	1.019 / (5/250)	1.452 / (6/250)	2.280 / (1/100)	4.752	zero

markedly as X increases. It is further observed that this decrease is relatively more when X increases from 8 to 12 and 16 to 20 than when X increases from 4 to 8 and 12 to 16. This was considered to be related to the shape and size of the quinine molecule and the pore size of the resin particle.

Elution of quinine from the column with N/10 sulphuric acid becomes less efficient as X increases. Same is true for elution with 1 N sulphuric acid. Elution with 1 N sulphuric acid is better, as may be expected, than with N/10 sulphuric acid. Elution of liberated quinine with ethanol also becomes less efficient as X increases. The lower efficiency of elution with X increasing, is due to reduced rate of diffusion through and from the resin particles as X increases.

The conclusion from the effect of degree of cross-linking is that lower degree of cross-linking is to be preferred, as this would permit higher break-through capacity due to higher rate of exchange and higher effective capacity of the resin. However if X is reduced below 4 , the volume changes during exchange would be relatively more and (molecular) sorption also would be operative (Section Four), which would be undesirable.

Resin IR-200 and IR-120 have been studied with different particle diameters . The uptake from both sodium sulphate solution and from quinine sulphate solution is markedly increased by decrease in particle size. The increase is more marked in case of quinine sulphate. This is in accordance with the observation (Section Six) that rate of exchange is increased with decrease in particle size. With resin IR-120, the break-through capacity as well as elution are poor and comparable to X 8. With IR-200, which is considerably more porous, though of X about 8, the rate of exchange is relatively higher than that for X 8, or IR-120 and hence the uptake and elution are relatively more efficient (both with sulphuric acid and ethanol). The elution is also better than that for X 4. However, the effective capacity for IR-200 is less than that for X 4 or X 8. The particle diameter has again a marked effect, on break-through capacity and on elution. The resin IR-200 is considered to have better thermal and mechanical stability.

The above indicates that a resin of relatively high cross-linking, such as X 20, would be suitable for removing small cations from a solution of alkaloid salt ; by passing the solution through the column, the small cations will be preferably taken up by the exchanger while most of the organic alkaloid cations will pass through. The acid in the effluent, then, may be removed with a weakly basic resin such as Amberlite IR-4 B .

On the other hand for recovery of alkaloid from raw material, the choice should be between X 4 and IR-200 , the particle size being small ; however, the pressure-drop would increase as the particle size is decreased. Hence a compromise has to be made. Further detailed work should indicate which of the two should be preferred on the over-all considerations.