## 7. EXTRACTION OF THE ALKALOIDS FROM CINCHONA BARK, CINCHONA FEBRIFUGE AND TOTAQUINA

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### 7.1

### Introduction :

This study includes work on extraction of the alkaloids from cinchona bark, cinchona febrifuge and totaquina using the resins Dowex 50W X4 and Amberlite-200.

### 7.2

### Cinchona bark :

### 7.2.1

### Experimental :

C.P.grade chemicals were used. Cinchona bark was supplied by the Government Quinine Factory, West Bengal. It was powdered (-60 mesh) and the alkaloid content was estimated (164) as follows :

Powdered cinchona bark (15 gm) was mixed with saturated lead subacetate solution (11.3 cc.) and water (18.8 cc.), allowed to stand for an hour, ammonical alcohol (75 cc.) added, mixed well, again allowed to stand for an hour, transferred to a soxhelt extractor with some more ammonical alcohol and extracted for four hours. Then most of the alcohol from the alcoholic extract was evaporated on a water bath, N sulfuric acid (15 cc.) and water (60 cc.) added, heated to boiling and cooled. The liquid was filtered through a tightly packed plug of cotton wool, previously

moistened with water, into a separator. The residue in the flask was treated with boiling N/10 sulfuric acid (20-30 cc.), cooled and filtered into the same separator. Finally the flask and the cotton wool were washed free of alkaloids with cold dilute aqueous sulfuric acid. Chloroform (30 cc.) was added to the solution in the separator and agitated for about two minutes. The chloroform layer was allowed to separate and run into a second separator containing N sulfuric acid (7.5 cc.) and water (22.5 cc.) and shaken well, the chloroform layer again allowed to separate and rejected. The liquid in the first separator was shaken with chloroform (30 cc.), run into the second separator and washed with dilute acid as before. This treatment of the liquid in the first separator was repeated. The acid washings were transferred to the first separator, excess of dilute ammonia solution was added and shaken with successive quantities of chloroform until complete extraction was effected. The chloroform solution of the alkaloids was washed with a little water, chloroform removed as completely as possible, distilled ethyl alcohol (7.5 cc.) added, evaporated to dryness on a water bath, dried at 100 $\pm$ 1°C and finally weighed. The amount of extracted alkaloids was 0.975 gm , that is 6.5 gm in 100 gm of the powdered bark. The extracted alkaloid material was then analysed as follows (164, 165) :

Two grams of the extracted alkaloid material were dissolved in N sulfuric acid (20 cc.), distilled ethyl alcohol (40 cc.) and water (40 cc.). This was heated to boiling ; N/10 sodium hydroxide solution was added, keeping

the liquid hot during the addition, until the solution was just faintly alkaline to solution of litmus. It was allowed to cool, N/10 sulfuric acid was added drop by drop, until the solution was slightly acidic to litmus. It was then boiled for about two minutes, cooled, again rendered slightly acidic to litmus, if necessary, boiled and filtered into a tared flask. The original vessel and the filter were washed with boiling water until complete extraction of the alkaloids was effected, adding the washings to the original filtrate. The filtrate was evaporated until it weighed about 120 gm . Powdered sodium potassium tartarate (30 gm ) was added and was allowed to stand for 24 hours. The precipitate was filtered through a hardened filter (Whatman No.1), the flask and the filter were washed with 25 % W/V solution (80 cc.) of sodium potassium tartarate in water, added in portions. The filtrate and washings were preserved. The filter with precipitate was transferred to the flask, 20 % sodium hydroxide (40 cc.) and chloroform (80 cc.) were added, allowed to stand, shaking from time to time, until complete solution was effected. The chloroform solution was separated, the flask and the aqueous liquid were washed with a little water. The chloroform layer was removed into a weighed flask, distilled ethyl alcohol (5 cc.) was added, evaporated on a water bath, dried at 100 $\pm$ 1°C and finally weighed. The difference in weights gave the amount of quinine and cinchonidine in the mixture. Distilled ethyl alcohol was added into the same flask, the mixture of quinine and cinchonidine was dissolved and the amounts of quinine and

cinchonidine were estimated by studying the ultraviolet absorption at 332 mμ. (115) after suitable dilution with ethyl alcohol.

The filtrate and washings preserved earlier were run into a separator containing ether (80 cc.) and 20 % W/V sodium hydroxide solution (20 cc.) and shaken well. The aqueous layer was run into a second separator, shaken with two further quantities of ether (80 cc.), each quantity of ether being returned to the first separator. The mixed etherial solution was washed with a little water and the alkaloids were extracted by shaking with successive quantities of N sulfuric acid (10, 10 and 5 cc.) and finally with water (10 cc.). The mixed acid and aqueous liquids were run into a separator containing ether (25 cc.) and N sodium hydroxide (30 cc.), shaken well and were allowed to stand for an hour. The precipitated cinchonine was collected on a tared filter, using a little water to facilitate the complete transfer of the precipitate to the filter ; the ether from the filter was separated, it was again run through the precipitate on the filter. The aqueous liquid was again shaken with two separate quantities of ether (25 cc.) and these etherial washings were used to wash the precipitate. This precipitate of cinchonine was dried at 100 ± 1°C and weighed. To this weight, 0.08 gm was added in order to correct for the loss of cinchonine due to its solubility in ether.

The etherial filtrate from the cinchonine was run into a separator, the filter flask was washed with a little

water and ether and washings were added to the liquid in the separator. The aqueous layer was separated and alkaloid from etherial solution was extracted by shaking with successive quantities of 10 % W/W aqueous solution of glacial acetic acid (10, 10, 5 and 5 cc.) which had been previously used to wash out any alkaloid remaining in the filter flask or on the stem of the funnel. The mixed acid solution was heated to boiling point, neutralised with dilute solution of ammonia, potassium iodide (5 gm ) was added, allowed to stand over night, the supernatant liquid was decanted through a filter paper, the precipitate was warmed with 50 % ethyl alcohol (5 cc.), the liquid was filtered off and 50 % ethyl alcohol (5 cc.) was passed through the crystalline residue. It was then dried at 100 $\pm$ 1°C and weighed. To the weight of quinidine hydriodide thus obtained, 0.008 gm was added to correct for the loss of quinidine hydriodide due to its solubility. Each gm of quinidine hydriodide is equivalent to 0.717 gm of quinidine.

Here, at the time of addition of potassium iodide to the solution of quinidine, care should be taken for proper neutralisation of the solution with dilute solution of ammonia. Add slowly dilute solution of ammonia until the solution is just neutral to litmus. Then only add potassium iodide to the neutral solution to precipitate quinidine hydriodide.

Theanalysis based on the alkaloid material was : quinine = 18 % ; quinidine = 17.5 % ; cinchonine = 29.0 % ; cinchonidine = 9.0 % ; other material (by difference) = 26.5 %.

Hence the result based on bark was :
quinine = 1.17 % ; quinidine = 1.14 % ; cinchonine = 1.88 % ; cinchonidine = 0.59 % ; other material = 1.72 % .

7.2.2

Results and Discussion :

To study the extraction of the alkaloid material from cinchona bark by ion exchange, 24 runs were carried out using columns of resins Dowex 50W X4 and Amberlite-200 in ammonium form.

Section I :

In this section, 12 runs were carried out using resin Dowex 50W X4 in ammonium form.

Run 1 :

A column of resin X4 (100/200 mesh) in ammonium form was set up. The data for the column were :
bed length = 44.1 cms ; bed volume = 75.0 cc. ; capacity of the resin in the column = 100 meq.

Powdered cinchona bark (77 gm ) was put into a beaker containing four litres of N/100 sulfuric acid (pH = 2.2 - 2.3), stirred well for four hours, allowed to stand over night and filtered. The filtrate was taken in an aspirator and passed through the column. The flow rate was about 10 cc. per minute. The effluent from the column was collected in a beaker, the pH was adjusted to the original value, returned to the bark container, stirred well and filtered. The filtrate was transferred to the aspirator and thus the whole process was continued till the alkaloids

were removed from the material as indicated by the absence of the fluorescence. The amount of acid solution passed through the column was 44 litres in about 74 hours. Then the column was washed with distilled water, backwashed to remove the resineous and other insoluble materials from the resin bed. 50 % aqueous ethyl alcohol (50 cc.) was passed through the column.

The alkaloid material was eluted by passing 6 litres (in about 67 hours) of N/10 ammonical ethyl alcohol at a flow rate of 1.5 cc. per minute through the column. The alcoholic extract of alkaloids was concentrated by distillation, evaporated on a water bath, dried at 100 ± 1°C and weighed. The amount of alkaloid material obtained was 5 gm . The analysis was :

quinine = 0.90 gm ; quinidine = 0.88 gm ; cinchonine = 1.45 gm ; cinchonidine = 0.45 gm ; other material = 1.32 gm .

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.

Runs 2 to 4 :

These were the repetition of run 1 except that acid used for extraction of alkaloids from cinchona bark was N/10 sulfuric acid for run 2, N/100 hydrochloric acid for run 3 and N/10 hydrochloric acid for run 4.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 1.

Run 5 :

This was the repetition of run 1 except that amount of powdered cinchona bark taken was 154 gm .

The amount of alkaloid material obtained was 10 gm . The analysis was :

quinine = 1.80 gm ; quinidine = 1.75 gm ; cinchonine = 2.90 gm ; cinchonidine = 0.90 gm ; other material = 2.65 gm. .

Runs 6 to 8 :

These were the repetition of run 5 except that acid used for extraction of alkaloids from cinchona bark was N/10 sulfuric acid for run 6, N/100 hydrochloric acid for run 7 and N/10 hydrochloric acid for run 8.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 5.

Run 9 :

This was the repetition of run 1 except that amount of powdered cinchona bark taken was 1000 gm . Also the cycle of extraction of the bark with aqueous acid solution and then exchange with the resin column was repeated till the alkaloid content of the influent and effluent was almost same as indicated by fluorescence.

The amount of alkaloid material obtained was 15 gm . The analysis was :

quinine = 2.70 gm ; quinidine = 2.63 gm ; cinchonine = 3.90 gm ; cinchonidine = 1.35 gm ; other material = 4.42 gm .

Runs 10 to 12 :

These were the repetition of run 9 except that acid used for extraction of alkaloids from cinchona bark was N/10 sulfuric acid for run 10, N/100 hydrochloric acid for run 11 and N/10 hydrochloric acid for run 12.

The amount and analysis of the alkaloid material obtained in each case were practically same as those of run 9.

Section II :

In this section, 12 runs were carried out using resin Amberlite-200 in ammonium form.

Run 13 :

A column of resin Amberlite-200 (20/50 mesh) in ammonium form was set up. The data for the column were : bed length = 35 cms ; bed volume = 59.5 cc. ; capacity of the resin in the column = 100 meq.

The procedure followed for this run was same as that given for run 1 except that amount of cinchona bark taken was 71 gm and the amount of acid solution (N/100 sulfuric acid) passed through the column was 24 litres in about 40 hours.

The alkaloid material was eluted by passing 3 litres (in about 34 hours) of N/10 ammonical ethyl alcohol at a flow rate of 1.5 cc. per minute through the column. The amount of alkaloid material obtained was 4.6 gm . The analysis was :
quinine = 0.83 gm ; quinidine = 0.81 gm ; cinchonine = 1.33 gm ; cinchonidine = 0.41 gm ; other material = 1.22 gm .

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.

Runs 14 to 16 :

These were the repetition of run 13 except that acid used for extraction of alkaloids from cinchona bark was N/10 sulfuric acid for run 14, N/100 hydrochloric acid for run 15 and N/10 hydrochloric acid for run 16.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 13.

Run 17 :

This was the repetition of run 13 except that amount of cinchona bark taken was 92.3 gm .

The amount of alkaloid material obtained was 5.7 gm . The analysis was :
quinine = 1.03 gm ; quinidine = 1.00 gm ; cinchonine = 1.54 gm ; cinchonidine = 0.51 gm ; other material = 1.62 gm .

Runs 18 to 20 :

These were the repetition of run 17 except that acid used for extraction of alkaloids from cinchona bark was N/10 sulfuric acid for run 18, N/100 hydrochloric acid for run 19 and N/10 hydrochloric acid for run 20.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 17.

Run 21 :

This was the repetition of run 13 except that amount of cinchona bark taken was 357 gm . Also the cycle of extraction of the bark with aqueous acid solution and then exchange with the resin column was repeated till the alkaloid content of the influent and effluent was almost same as indicated by fluorescence.

The amount of alkaloid material obtained was 8 gm . The analysis was :

quinine = 1.44 gm ; quinidine = 1.40 gm ; cinchonine = 1.60 gm ; cinchonidine = 0.72 gm ; other material = 2.84 gm .

Run 22 :

This was the repetition of run 21 except that acid used for extraction of alkaloids from cinchona bark was N/100 hydrochloric acid.

The amount and analysis of alkaloid material obtained were practically same as those of run 21.

Runs 23 to 24 :

These were the repetition of run 21 except that acid used for the extraction of alkaloids from cinchona bark was N/10 sulfuric acid for run 23 and N/10 hydrochloric acid for run 24. Also in both the runs, 500 cc. of 50 % aqueous ethyl alcohol in place of 50 cc. of the same was passed through the column before the elution was started.

The amount of alkaloid material obtained was 6.9 gm .

Table 7.1

Summary of the data of runs 1 to 24.

The first line gives the results obtained by solvent extraction.

| Run No. | Amount of Bark. ( gm ) | Y* ( gm ) | Amount of ( gm ) | | | | | % of | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | QN | CND | CN | QND | Others | QN | CND | CN | QND | Others |
| - | 100 | 6.5 | 1.17 | 0.59 | 1.88 | 1.14 | 1.72 | 18.0 | 9.0 | 29.0 | 17.5 | 26.5 |
| 1 | 77 | 5.0 | 0.90 | 0.45 | 1.45 | 0.88 | 1.32 | 18.0 | 9.0 | 29.0 | 17.5 | 26.5 |
| 5 | 154 | 10.0 | 1.80 | 0.90 | 2.90 | 1.75 | 2.65 | 18.0 | 9.0 | 29.0 | 17.5 | 26.5 |
| 9 | 1000 | 15.0 | 2.70 | 1.35 | 3.90 | 2.63 | 4.42 | 18.0 | 9.0 | 26.0 | 17.5 | 29.5 |
| 13 | 71 | 4.6 | 0.83 | 0.41 | 1.33 | 0.81 | 1.22 | 18.0 | 9.0 | 29.0 | 17.5 | 26.5 |
| 17 | 92.3 | 5.7 | 1.03 | 0.51 | 1.54 | 1.00 | 1.62 | 18.0 | 9.0 | 27.0 | 17.5 | 28.5 |
| 21 | 357 | 8.0 | 1.44 | 0.72 | 1.60 | 1.40 | 2.84 | 18.0 | 9.0 | 20.0 | 17.5 | 35.5 |
| 23 | 357 | 6.9 | 1.24 | 0.69 | 1.38 | 0.76 | 2.83 | 18.0 | 10.0 | 20.0 | 11.0 | 41.0 |

QN = quinine, CND = cinchonidine, CN = cinchonine, QND = quinidine.

Y = amount of alkaloid material obtained.

The analysis was :

quinine = 1.24 gm ; quinidine = 0.76 gm ; cinchonine = 1.38 gm ; cinchonidine = 0.69 gm ; other material = 2.83 gm .

The results of the above runs indicate that other conditions being same, the results are practically same with sulfuric acid and hydrochloric acid for concentrations N/10 and N/100.

Results of the runs (1 to 24) are summarised in Table 7.1. When the weight of the bark is relatively much less as compared to the capacity of the resin in the column, the extraction of the alkaloids is practically complete and the composition of the extracted material is also essentially same as that for material obtained by solvent extraction. When the amount of the bark taken is relatively in large excess as compared to the capacity of the resin in the column, the amount of extracted alkaloids is 15 gm for resin X4 and 8 gm for resin Amberlite-200, capacity of the resin in the column being 100 meq. This indicates that practically all of the effective capacity has been used (Chapter 3). The composition of the extracted material indicate that, under these conditions, the percentage of cinchonine in the extracted material is less, by 3 % for X4 and 9 % for Amberlite-200, than that obtained by solvent extraction.

The result of run 23 indicates that when 500 cc., instead of 50 cc., of 50 % aqueous ethyl alcohol were used for washing the column before elution, the amount of extracted material is reduced to 6.9 gm from 8 gm .

In the percentage composition the percent of quinidine has also been reduced.

7.3

Cinchona febrifuge :

7.3.1

Experimental :

Cinchona febrifuge was supplied by the Government Quinine Factory, West Bengal. Samples (2 gm ) were analysed by procedure similar to that given for the analysis of cinchona bark.

The analysis was :

quinine = 0.43 gm ; quinidine = 0.46 gm ; cinchonine = 0.19 gm ; cinchonidine = 0.13 gm ; other material (by difference)= 0.79 gm

The percent analysis was :

quinine = 21.5 % ; quinidine = 23.0 % ; cinchonine = 9.5 % ; cinchonidine = 6.5 % ; other material = 39.5 % .

7.3.2

Results and Discussion :

To study the extraction of the alkaloid material from cinchona febrifuge by ion exchange, 24 runs were carried out using columns of resins Dowex 50W X4 and Amberlite-200 in ammonium form.

Section I :

In this section, 12 runs were carried out using resin Dowex 50W X4 in ammonium form.

Run 25 :

A column of resin X4 (100/200 mesh) in ammonium form was set up as given for run 1.

Cinchona febrifuge (8 gm ) was taken and procedure similar to that given for run 1 was followed using N/100 sulfuric acid as extraction solvent. The amount of acid solution passed through the column was 18 litres in about 30 hours.

The alkaloid material was eluted by passing 6 litres (in about 67 hours) of N/10 ammonical ethyl alcohol at a flow rate of 1.5 cc. per minute through the column. The amount of alkaloid material obtained was 6 gm . The analysis was :

quinine = 1.72 gm ; quinidine = 1.84 gm ; cinchonine = 0.76 gm ; cinchonidine = 0.52 gm ; other material = 1.16 gm .

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 runs.

Runs 26 to 28 :

These were the repetition of run 25 except that acid used for extraction of alkaloids from cinchona febrifuge was N/10 sulfuric acid for run 26, N/100 hydrochloric acid for run 27 and N/10 hydrochloric acid for run 28.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 25.

Run 29 :

This was the repetition of run 25 except that amount of cinchona febrifuge taken was 16 gm .

The amount of alkaloid material obtained was 12 gm . The analysis was :

quinine = 3.44 gm ; quinidine = 3.68 gm ; cinchonine = 1.52 gm ; cinchonidine = 1.04 gm ; other material = 2.32 gm .

Runs 30 to 32 :

These were the repetition of run 29 except that acid used for extraction of alkaloids from cinchona febrifuge was N/10 sulfuric acid for run 30, N/100 hydrochloric acid for run 31 and N/10 hydrochloric acid for run 32.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 29.

Run 33 :

This was the repetition of run 25 except that amount of cinchona febrifuge taken was 44 gm . Also the cycle of extraction of the febrifuge with aqueous acid solution and then exchange with the resin column was repeated till the alkaloid content of the influent and effluent was almost same as indicated by fluorescence.

The amount of alkaloid material obtained was 15 gm . The analysis was :

quinine = 4.80 gm ; quinidine = 4.80 gm ; cinchonine = 1.28 gm ; cinchonidine = 0.90 gm ; other material = 3.22 gm .

Runs 34 to 36 :

These were the repetition of run 33 except that acid used for extraction of alkaloid from cinchona febrifuge was N/10 sulfuric acid for run 34, N/100 hydrochloric acid for run 35 and N/10 hydrochloric acid for run 36.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 33.

Section II :

In this section, 12 runs were carried out using resin Amberlite-200 in ammonium form.

Run 37 :

A column of resin Amberlite-200 (20/50 mesh) in ammonium form was set up as given for run 13.

The procedure followed for this run was same as that given for run 25 except that amount of cinchona febrifuge taken was 5.6 gm and the amount of acid solution (N/100 sulfuric acid) passed through the column was 10 litres in about 17 hours.

The alkaloid material was eluted by passing 3 litres (in about 34 hours) of N/10 ammonical ethyl alcohol at a flow rate of 1.5 cc. per minute through the column. The amount of alkaloid material obtained was 4.2 gm . The analysis was :

quinine = 1.20 gm ; quinidine = 1.29 gm ; cinchonine = 0.53 gm ; cinchonidine = 0.37 gm ; other material = 0.81 gm .

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.

Runs 38 to 40 :

These were the repetition of run 37 except that acid used for extraction of alkaloids from cinchona febrifuge was N/10 sulfuric acid for run 38, N/100 hydrochloric acid for run 39 and N/10 hydrochloric acid for run 40.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 37.

Run 41 :

This was the repetition of run 37 except that amount of cinchona febrifuge taken was 10.4 gm .

The amount of alkaloid material obtained was 7.4 gm . The analysis was :

quinine = 2.24 gm ; quinidine = 2.39 gm ; cinchonine = 0.59 gm ; cinchonidine = 0.68 gm ; other material = 1.50 gm .

Runs 42 to 44 :

These werevthe repetition of run 41 except that acid used for extraction of alkaloids from cinchona febrifuge was N/10 sulfuric acid for run 42, N/100 hydrochloric acid for run 43 and N/10 hydrochloric acid for run 44.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 41.

Run 45 :

This was the repetition of run 37 except that

amount of cinchona febrifuge taken was 32 gm . Also the cycle of extraction of the febrifuge with aqueous acid solution and then exchange with the resin column was repeated till the alkaloid content of the influent and effluent was almost same as indicated by fluorescence.

The amount of alkaloid material obtained was 8 gm . The analysis was :
quinine = 2.64 gm ; quinidine = 2.64 gm ; cinchonine = 0.53 gm ; cinchonidine = 0.48 gm ; other material = 1.71 gm .

Runs 46 to 48 :

These were the repetition of run 45 except that acid used for extraction of alkaloids from cinchona febrifuge was N/10 sulfuric acid for run 46, N/100 hydrochloric acid for run 47 and N/10 hydrochloric acid for run 48.

The amount and analysis of the alkaloid material obtained in each case were practically same as those of run 45.

Results of the runs (25 to 48) are summarised in Table 7.2. Runs indicate that the results are practically same for hydrochloric acid and sulfuric acid of concentrations N/10 and N/100 as extraction solvents.

The result of run 25 indicates that about $1/4^{th}$ of the weight of the febrifuge used is not extracted by the resin ; and, hence, is probably not basic. However, weight of quinine, quinidine, cinchonine and cinchonidine is practically same as may be expected from the analysis by solvent extraction. Hence, the percentage of the cinchona

Table 7.2

Summary of the data of runs 25 to 48.

The first line gives the results obtained by solvent extraction.

| Run No. | Amount of Febrifuge. ( gm ) | Y ( gm ) | Amount of ..... ( gm ) | | | | | % of ..... | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | QN | CND | CN | QND | Others | QN | CND | CN | QND | Others |
| - | 2.0 | - | 0.43 | 0.13 | 0.19 | 0.46 | 0.79 | 21.5 | 6.5 | 9.5 | 23.0 | 39.5 |
| 25 | 8.0 | 6.0 | 1.72 | 0.52 | 0.76 | 1.84 | 1.16 | 28.7 | 8.7 | 12.7 | 30.7 | 19.2 |
| 29 | 16.0 | 12.0 | 3.44 | 1.04 | 1.52 | 3.68 | 2.32 | 28.7 | 8.7 | 12.7 | 30.7 | 19.2 |
| 33 | 44.0 | 15.0 | 4.80 | 0.90 | 1.28 | 4.80 | 3.22 | 32.0 | 6.0 | 8.5 | 32.0 | 21.5 |
| 37 | 5.6 | 4.2 | 1.20 | 0.37 | 0.53 | 1.29 | 0.81 | 28.6 | 8.8 | 12.6 | 30.7 | 19.3 |
| 41 | 10.4 | 7.4 | 2.24 | 0.68 | 0.59 | 2.39 | 1.50 | 30.3 | 9.2 | 8.0 | 32.3 | 20.2 |
| 45 | 32.0 | 8.0 | 2.64 | 0.48 | 0.53 | 2.64 | 1.71 | 33.0 | 6.0 | 6.6 | 33.0 | 21.4 |

QN = quinine, CND = cinchonidine, CN = cinchonine, QND = quinidine.

Y = amount of alkaloid material obtained.

alkaloids in extracted material will be different from that given on the basis of solvent extraction. This is based on the reasonable assumption that there is practically complete extraction of extractable material, when amount of cinchona febrifuge used is relatively much less than the capacity of the resin in the column. The results of run 29 are consistent with above conclusion. When the amount of cinchona febrifuge taken is relatively in large excess (run 33), the amount of extracted material obtained is 15 gm , which is almost the amount to be expected from the effective capacity of the column.

The percentage of quinine and quinidine in extracted material are higher than those obtained from solvent extraction. This should imply that, by ion exchange treatment, the percent cinchona alkaloid content of cinchona febrifuge is improved.

The results with resin Amberlite-200 are consistent with this conclusion. The amount extracted, when relatively large excess of cinchona febrifuge is used, was 8 gm for 100 meq. of resin.

7.4

Totaquina :

7.4.1

Experimental :

Totaquina was supplied by the Government Quinine Factory, West Bengal. Samples (2 gm ) were analysed by

procedure similar to that given for the analysis of cinchona bark.

The analysis was :

quinine = 0.58 gm ; quinidine = 0.60 gm ; cinchonine = 0.29 gm ; cinchonidine = 0.21 gm ; other material (by difference)≒ 0.32 gm

The percent analysis was :

quinine = 29.0 % ; quinidine = 30.0 % ; cinchonine = 14.5 % ; cinchonidine = 10.5 % ; other material = 16.0 % .

7.4.2

Results and Discussion :

To study the extraction of the alkaloid material from totaquina by ion exchange, 28 runs were carried out using columns of resins Dowex 50W X4 and Amberlite-200 in ammonium form.

Section I :

In this section, 12 runs were carried out using resin Dowex 50W X4 in ammonium form.

Run 49 :

A column of resin X4 (100/200 mesh) in ammonium form was set up as given for run 1.

Totaquina (8 gm ) was taken and procedure similar to that given for run 1 was followed using N/100 sulfuric acid as extraction solvent. The amount of acid solution passed through the column was 18 litres in about 30 hours.

The alkaloid material was eluted by passing 6 litres (in about 67 hours) of N/10 ammonical ethyl alcohol at a

flow rate of 1.5 cc. per minute through the column. The amount of alkaloid material obtained was 7.2 gm . The analysis was :

quinine = 2.32 gm ; quinidine = 2.40 gm ; cinchonine = 1.16 gm ; cinchonidine = 0.84 gm ; other material = 0.48 gm .

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.

Runs 50 to 52 :

These were the repetition of run 49 except that acid used for extraction of alkaloids from totaquina was N/10 sulfuric acid for run 50, N/100 hydrochloric acid for run 51 and N/10 hydrochloric acid for run 52.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 49.

Run 53 :

This was the repetition of run 49 except that amount of totaquina taken was 13 gm .

The amount of alkaloid material obtained was 11.7 gm . The analysis was :

quinine = 3.77 gm ; quinidine = 3.90 gm ; cinchonine = 1.88 gm ; cinchonidine = 1.37 gm ; other material = 0.78 gm .

Runs 54 to 56 :

These were the repetition of run 53 except that acid used for extraction of alkaloids from totaquina was

N/10 sulfuric acid for run 54, N/100 hydrochloric acid for run 55 and N/10 hydrochloric acid for run 56.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 53.

Run 57 :

This was the repetition of run 49 except that amount of totaquina taken was 44 gm . Also the cycle of extraction of totaquina with aqueous acid solution and then exchange with the resin column was repeated till the alkaloid content of the influent and effluent was almost same as indicated by fluorescence.

The amount of alkaloid material obtained was 15 gm . The analysis was :

quinine = 4.83 gm ; quinidine = 5.00 gm ; cinchonine = 2.02 gm ; cinchonidine = 1.58 gm ; other material = 1.57 gm .

Runs 58 to 60 :

These were the repetition of run 57 except that acid used for extraction of alkaloids from totaquina was N/10 sulfuric acid for run 58, N/100 hydrochloric acid for run 59 and N/10 hydrochloric acid for run 60.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 57.

Section II :

In this section, 16 runs were carried out using resin Amberlite-200 in ammonium form.

Run 61 :

A column of resin Amberlite-200 (20/50 mesh) in ammonium form was set up as given for run 13.

The procedure followed for this run was same as that given for run 37 except that amount of totaquina taken was 5.6 gm and the amount of acid solution (N/100 sulfuric acid) passed through the column was 10 litres in about 17 hours.

The alkaloid material was eluted by passing 3 litres (in about 3½ hours) of N/10 ammonical ethyl alcohol at a flow rate of 1.5 cc. per minute through the column. The amount of alkaloid material obtained was 5 gm . The analysis was :

quinine = 1.62 gm ; quinidine = 1.68 gm ; cinchonine = 0.81 gm ; cinchonidine = 0.59 gm ; other material = 0.30 gm .

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.

Runs 62 to 64 :

These were the repetition of run 61 except that acid used for extraction of alkaloids from totaquina was N/10 sulfuric acid for run 62, N/100 hydrochloric acid for run 63 and N/10 hydrochloric acid for run 64.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 61.

Run 65 :

This was the repetition of run 61 except that amount of totaquina taken was 7.2 gm .

The amount of alkaloid material obtained was 6.4 gm . The analysis was :

quinine = 2.09 gm ; quinidine = 2.16 gm ; cinchonine = 0.96 gm ; cinchonidine = 0.76 gm ; other material = 0.43 gm .

Runs 66 to 68 :

These were the repetition of run 65 except that acid used for extraction of alkaloids from totaquina was N/10 sulfuric acid for run 66, N/100 hydrochloric acid for run 67 and N/10 hydrochloric acid for run 68.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 65.

Run 69 :

This was the repetition of run 65 except that amount of totaquina taken was 8.6 gm .

The amount of alkaloid material obtained was 7.4 gm . The analysis was :

quinine = 2.49 gm ; quinidine = 2.58 gm ; cinchonine = 0.99 gm ; cinchonidine = 0.82 gm ; other material = 0.52 gm .

Runs 70 to 72 :

These were the repetition of run 69 except that acid used for extraction of alkaloids from totaquina was N/10 sulfuric acid for run 70, N/100 hydrochloric acid for run 71 and N/10 hydrochloric acid for run 72.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 69.

Run 73 :

This was the repetition of run 61 except that amount of totaquina taken was 30 gm . Also the cycle of extraction of totaquina with aqueous acid solution and then exchange with the resin column was repeated till the alkaloid content of the influent and effluent was almost same as indicated by fluorescence.

The amount of alkaloid material obtained was 8 gm . The analysis was :

quinine = 2.80 gm ; quinidine = 2.80 gm ; cinchonine = 0.80 gm ; cinchonidine = 0.84 gm ; other material = 0.76 gm .

Runs 74 to 76 :

These were the repetition of run 73 except that acid used for extraction of alkaloids from totaquina was N/10 sulfuric acid for run 74, N/100 hydrochloric acid for run 75 and N/10 hydrochloric acid for run 76.

The amount and analysis of alkaloid material obtained in each case were practically same as those of run 73.

Results of the runs (49 to 76) are summarised in Table 7.3 . The runs again indicate that results are not significantly different when the extraction solvent is aqueous hydrochloric acid or sulfuric acid of concentrations N/10 and N/100.

The results of run 49 indicate that about 10 % of the totaquina used, is not extractable by ion exchange method.

Table 7.3

Summary of the data of runs 49 to 76.

The first line gives the results obtained by solvent extraction.

| Run No. | Amount of Totaquina. ( gm ) | Y ( gm ) | Amount of ( gm ) QN | CND | CN | QND | Others | % of QN | CND | CN | QND | Others |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| - | 2.0 | - | 0.58 | 0.21 | 0.29 | 0.60 | 0.32 | 29.0 | 10.5 | 14.5 | 30.0 | 16.0 |
| 49 | 8.0 | 7.2 | 2.32 | 0.84 | 1.16 | 2.40 | 0.48 | 32.2 | 11.7 | 16.1 | 33.3 | 6.7 |
| 53 | 13.0 | 11.7 | 3.77 | 1.37 | 1.88 | 3.90 | 0.78 | 32.2 | 11.7 | 16.1 | 33.3 | 6.7 |
| 57 | 44.0 | 15.0 | 4.83 | 1.58 | 2.02 | 5.00 | 1.57 | 32.2 | 10.5 | 13.5 | 33.3 | 10.5 |
| 61 | 5.6 | 5.0 | 1.62 | 0.59 | 0.81 | 1.68 | 0.30 | 32.4 | 11.8 | 16.2 | 33.6 | 6.0 |
| 65 | 7.2 | 6.4 | 2.09 | 0.76 | 0.96 | 2.16 | 0.43 | 32.7 | 11.9 | 15.0 | 33.7 | 6.7 |
| 69 | 8.6 | 7.4 | 2.49 | 0.82 | 0.99 | 2.58 | 0.52 | 33.6 | 11.1 | 13.4 | 35.0 | 6.9 |
| 73 | 30.0 | 8.0 | 2.80 | 0.84 | 0.80 | 2.80 | 0.76 | 35.0 | 10.5 | 10.0 | 35.0 | 9.5 |

QN = quinine, CND = cinchonidine, CN = cinchonine, QND = quinidine.

Y = amount of alkaloid material obtained.

This is based on the reasonable assumption that there is a complete extraction of extractable material, when amount of totaquina used is much less than capacity of the resin in the column. This is consistent with the results of run 53.

When relatively large amount of totaquina was used, the amount of extractable material was 15 gm and percentage of quinine and quinidine in the extracted material were increased and that of cinchonine has been decreased. Similar results were obtained with resin Amberlite-200. Also the amount extracted was 8 gm for 100 meq. of the resin.

The runs indicate that the results of both the resins are fairly comparable. Since resin Amberlite-200 has a better stability than resin X4, because of its higher degree of crosslinking and expanded structure, resin Amberlite-200 may be preferred for extraction of alkaloids from raw materials, using the resin in ammonium form with dilute aqueous sulfuric acid as extraction solvent and ammonical ethyl alcohol as an eluant. It should be desirable to access the stability of the resins for repeated use over a period of time.