## A Suggested Method for the Extraction of Turpentine, Resin and Gum from the Gum-oleo-resin of Boswellia Serrata without the use of Solvents.

By Gilbert J. Fowler and Mangesh Anant Malandkar.

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### INTRODUCTION.

The work described in the present paper was undertaken at the request of Mr. R. S. Pearson, Forest Economist, who, in conjunction with Mr. Puran Singh, has published a Memoir entitled "Note on the preparation of Turpentine, Resin and Gum, from *Boswellia Serratta* (Roxb.) gum-oleo-resin\*" in which very complete information is given as to the occurrence, mode of collection, chemistry and technical handling of Boswellia gum-oleo-resin.

Certain problems in connection particularly with the technical working up of the products *viz*. turpentine, resin and gum remained to be dealt with and it was thought that suitable equipment existed at the Institute of Science for their solution.

One of two processes of extraction are suggested by Pearson and Puran Singh.

- (i) Solvent extraction followed by steam distillation.
- (ii) Steam-distillation followed by solvent extraction.

Of the two the latter seems most likely to be satisfactory.

The solvents recommended are petroleum benzine or trichloroethylene. Both of these are less easily obtainable in India than alcohol and consequently in taking up the problem afresh in the experiments to be recorded in this paper, alcohol was first tried as a solvent.

Preliminary laboratory tests revealed certain difficulties. The removal of turpentine by vacuum distillation left a residue of dehydrated gum and resin which could be melted and handled

<sup>\*</sup> Indian Forest Records, Vol. VI, Part VI.

only with difficulty. If the turpentine is driven off with moderately superheated steam, this dehydration does not take place and if wet steam is used a highly hydrated product is obtained which melts quite readily. The expulsion of the turpentine by some form of steam distillation is thus indicated.

The observation that the oleo-resin tended to collect at the bottom of the tins in which it was stored led to the suggestion that it might be extracted by a simple melting process.

Experiment showed however that although a fair percentage of good material could be extracted in this way, the residue would still contain much resin and turpentine along with the gum and would again require separate treatment.

On boiling gum, from which the resin had been imperfectly separated, with water, it was found that the resin melted and rose to the surface from which it could be skimmed off. In an attempt to dissolve the gum by heating with water under pressure it was observed that under these conditions a practically complete separation of the gum and resin could be effected.

These experiments led finally to the method described in the following paper and which is suggested as probably more economical and easy to carry out than a solvent process, viz. the removal of the turpentine by wet steam, the cooking of the residue of hydrated gum resin under pressure with water when the hydrated resin collects together as a molten mass, leaving the gum as a flocculent mucilage. By evaporation, dehydration and sieving both resin and gum are obtained in a reasonably pure condition. The details of the experimental work are given in the following pages.

### PRELIMINARY EXAMINATION.

Some ten pounds of the crude gum-oleo-resin were first sent for examination and afterwards ten tins each containing about 35 lbs.

Preliminary analyses of four samples gave the following percentage results :---

Sample.	Moisture.	Turpentine.	Resin.	Gum, &c.
1	10.4	9.4	59.1	2].]
2	8.3	8.1	57.8	· 25·9
3	8.4	8.3	57-7	25 6
4	6.1	7.8	57.1	29.0
Average.	8.3	8.4	57.9	25.4

Laboratory experiments on method of extraction by steam distillation followed by solvent extraction.

As this was the method which the experimental work at Dehra Dun suggested as most suitable and preferable for various reasons to extraction with solvents prior to steam distillation, some laboratory trials were made with quantities of about 150 grams.

The gum-oleo-resin was placed in a round bottomed flask which was heated in an oil bath, the temperature of which was kept in the neighbourhood of 125°C. Steam was passed through and the turpentine was readily distilled off. The residue in the flask after removal of the turpentine was evidently somewhat hydrated and could be easily melted out of the flask.

Portions of this residue containing resin, gum and mineral and woody extraneous matter were taken for extraction with various possible solvents for the sake of comparison. Ethyl alcohol (90%) butyl alcohol and carbon tetrachloride were used with the following results.

Solven <b>t</b> .	Yield of resin. Calculated on raw material.	Appearance of resin.
90% alcohol	59%	Brown and trans- parent.
Butyl alcohol	59%	do
Carbon tetrachloride.	69%	Dark and opaque.

The resin extracted by carbon tetrachloride left a dark residue on redissolving in 90% alcohol, even on repeated boiling. It is thus evident that the carbon tetrachloride extracts something additional to the resin, probably some extractive matter from the woody residues present.

The butyl alcohol was satisfactory but needs a higher temperature than ordinary alcohol. It can be readily driven off from the residual gum by means of steam and can be separated from the condensed water and if it can be cheaply obtained as a by-product of the acetone fermentation process its use as a solvent is worth consideration.

Some of the extractions were carried out in a copper Soxhlet extractor and it was noticed that in this case the resin obtained was darker than when glass apparatus was used and was found to contain traces of copper. This tendency of an alcoholic solution of a resin to take up copper has been noted by one of us in working with lac varnish on the large scale. The metal plant for such work should always be of tinned copper or better of

On no account must the solution come in contact aluminium. with iron.

The gum was found to hold up alcohol equal to about its own weight, and its recovery is not an easy matter. Steaming means expensive fractionation in order to recover high strength alcohol. Probably a current of warm air is the most satisfactory method for removing the alcohol which can be separated from the air by condensation in a manner similar to that employed on the large scale for recovering acetone from cordite.

Doubtless petroleum benzine as recommended in Pearson and Puran Singh's Memoir, apart from its inflammability is a more satisfactory solvent in many ways. The resin it yields is much paler than that obtained by the use of alcohol. Chlorinated hydrocarbons require that all parts of the plant shall be carefully tinned, and both types of solvent are much more difficult to obtain in India than alcohol and for this reason the experiments were conducted with this solvent.

Whatever type of solvent is employed the inevitable loss occuring during the process is one of the chief items of cost. Ιt appeared therefore that, if a method would be devised dispensing with the use of solvents it could probably be simple and cheaper assuming that the same quality and quantity of end products was turned out.

Inasmuch as experiments to be [described below show that practical results can be obtained without the use of any solvent, the detail of the solvent problem was not further pursued.

The following is a description together with the chief analytical constants of the turpentine, resin and gum obtained by distillation with superheated steam followed by extraction with 90% alcohol.

Turpentine. The oil was of a faint yellowish type specific gravity at 25°C=0.8470. Specific rotation  $[\infty]_p^5 = +5^{\circ}$ 36'

Fractional distillation of the oil dried over fused calcium chloride gave the following results :---

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(1)	82.8%	6 of the	oil passed below	160°C.
(2)	8.7	"	., between	160°C & 180°C
(3)	8.5	••	above 1	80°C

,,

above 180°C.

Fraction (1) was almost colourless.

" (2) very faint yellow.

,, (3) distinctly yellow.

*Resin.* As mentioned above, the resin obtained by extraction in a copper soxhlet was somewhat dark in colour. Its chief analytical constants were determined as follows:—

For comparison, figures obtained for resin obtained by the use of petroleum benzine are also given.

1	Resin by <b>alc</b> ohol	Resin by benzine.
Saponification value	71.4	68·9
Acid value	47.5	51.4
Ester value (by diff)	23.9	17.5
Iodine value (Wij's method)	115 <sup>.</sup> 0	103.2

Gum. This was obtained as a white powder mixed with small lumps which could be reduced to powder by gentle crushing when the whole could be separated from pieces of bark &c. by sieving.

This gum formed a mucilage with water, of indifferent sticking power. It did not actually dissolve in any quantity. When boiled with a moderately strong solution of caustic soda or carbonate of soda partial solution or emulsion took place and on addition of acid a gelatinous precipitate was thrown down which was again practically insoluble and impossible to filter.

Separation by melting out oleo-resin. During a discus. sion of these results it was suggested that it might be possible simply to melt out the oleo-resin without the use of solvents at This suggestion was supported by the fact that the oleo-resin all. tended to accumulate to some extent at the bottom of a tin of raw material. Accordingly an attempt was made on the laboratory scale to accelerate this process and melt out the oleo-resin from the gum. For this purpose the apparatus used consisted of two concentric hollow cylinders, one inside the other The inner cylinder which was 8" high and 3" in diameter could be drained out by means of a tube passing through the wall of the outer cylinder and provided with a tap. The upper end of the inner cylinder was closed by a large Indiarubber stopper through which was passed the stem of a reflux condenser. The space between the cylinders was filled with water or oil which could be heated.

The gum-oleo-resin was suspended in the inner cylinder by means of a wire gauze cage or muslin bag. In the experiments, the results of which are tabulated below, 150 grams of crude gum oleo-resin were taken. The percentage of oleo-resin drained out is calculated on the crude material. In the last column is given the actual percentage of oleo-resin in the crude gum-oleo-resin as determined by solvent analysis. Different meshes of strainer were used as follows:---

In Experiments 1, 4, 7, 10, a cylindrical coarse linen bag (diameter 2.5" height 3.3")

In Experiments 2, 5, 8, 11, a cylindrical 80 mesh per sq. in wire gauze cage (diameter 2.5" height 3")

In Experiments 3, 6, 9, 12, a cylindrical 20 mesh per sq in wire gauze cage (diameter 2.5" height 3")

No.	Oleo-resin drained in 4 hours.	Oleo-resin drained in 7 hours.	Nature of oleo-resin drained.	Percentage of oleo-resin in the gam-oleo-resin.
	Draining		ns a <b>t</b> the boiling tem- re o <b>f wa</b> ter.	
1	26%	32%	Practically free from gum.	64%
2	27%	32%	do	"
3	29%	35%	Mixed with some gum	• >7
	D	raining of top	portions at 130°C.	•
4	40%	<b>40%</b>	Practically free from gum.	"
5	40%	40%	$d\mathbf{o}$	71
6	42%	43%	Mixed with some gum	• •,
	Drain		portions at the boiling of water.	
7	. 28%	31%	Practically free from gum.	81%
8	28%	32%	do	,,
9	30%	34%	Mixed with some gun	
	Dr	aining of bott	om portion at 130°C.	
10	41%	41%	Practically free from gum.	n "
11	41%	42%	do	21
12	42%	44%	Mixed with some gu	m.
			<b>U</b>	,,

It will be seen that little more than half of the oleo-resin could be obtained in this way so that further treatment by solvents or otherwise would be required for the residue.

Unless therefore the resin melted out in this way was of distinctly higher quality than the product obtained by other methods, this process does not appear to possess any advantage.

Some of the above experiments were performed in a tin apparatus and others in a copper one. The drained oleo-resin from the copper apparatus was darker in colour than that from the tin. Copper was detected in the ash of the oleo-resin from the copper apparatus.

Turpentine could be recovered both from the drained portion aud the residue when they were subjected to steam distillation.

There was practically no increase in the amount of oleoresin drained out, when the 80 mesh wire gauze cage was made longer and narrower.

An attempt was made to determine whether the turpentine could be removed by distillation and the resin could be separated by draining in one and the same operation. Accordingly steam was passed into the apparatus from below the gum oleo-resin, suspended in the 80 mesh wire gauze cage, the temperature of the oil in the annular space being kept at  $130^{\circ}$ C. Turpentine came over with the steam but very slowly and the resin was found to have carried some gum with it.

Another method that suggested itself was to separate the turpentine first under reduced pressure and then melt out the resin from the residue. The experiment was tried in a flask heated on the oil bath, the pressure being reduced to 18 c. m. of mercury. Water and turpentine distilled over forming separate layers in the distillate. The results are tabulated below :---

Temperature of bath.	Yield of turpentine.	Time for distillation.	Yield of water.	Sp. Gr. of Turpentine.	Sp. Rotation of turpentine
120°C	8%	$4\frac{1}{2}$ hrs.	7%	0.8418 at 27°C.	+ 7°52'
130°C	8%	2 ,,	7%	0·8437 at 27·6°C.	+ 2°47'
140°C	8%	11,,	6%	0 8472 at 27.6°C.	- 2°32′

The turpentines obtained were yellowish in tinge and possessed a smell different from that of steam distilled turpentine.

The residue of gum and resin remaining behind could not be melted out of the flask so easily as the hydrated mixture of gum and resin remaining after steam distillation.

An average sample of this residue, when cooked with water in the autoclave yielded resin as hydrated resin and the gum formed a thick mucilage. The hydrated resin after dehydration was found to contain some matter insoluble in 90% alcohol but the quantity was not large.

For want of sufficient material, the draining out of resin from the above mixture of gum and resin left behind after the turpentine was distilled under reduced pressure, was not tried.

An attempt was, therefore, made to melt out the resin and distil off the turpentine under reduced pressure in one and the same operation. Accordingly the gum-oleo-resin was suspended in a linen bag in the jacketed copper cylinder already described. The temperature of the oil was kept at 140 C and the pressure was 18 cm of mercury. Resin equal to about 16% of the gum oleo-resin was melted out in this way.

By none of these methods is a clean separation effected between gum and resin.

It was evident from the observations made that the hydrated resin resulting from steam distillation of the turpentine was much easier to handle than resin simply melted in vacuo.

It is also of interest that the turpentines obtained by this method, showed a tendency to increase in specific gravity and change the sign of rotation on keeping. They also developed a yellow tinge, the intensity of colour being the greatest in the case of sample A (below) and least in the case of sample C. Steam distilled turpentine also showed the above changes but to a very small extent as will be seen from the following table.

In the following table :---

Sample A	is turpentine obtained when the temperature of the bath $=$	120°C.
Sample B	do	130°C.
Sample C	do	140°C.
Sample D	is ste <b>a</b> m distilled turpentine.	

				$[\infty]_{D}$		$[\mathbf{\infty}]_{\mathrm{D}}$
Sample.	Specific gravity.	Specific rotation. $[\infty]_{D}$	Sp. Gra. at 30°C on 15th July 1920.	Sp. rotation on the 15th July 1920.	Sp. Gra. at 30°C on the 23rd Octr. 1920.	Sp. rotation on the 23rd Octr. 1920.
А.	0.8418 at 27°C on the 16th March 1920.	+ 7°52′ on the 16th March 1920.	0.9111	— 20°23′	0.9369	— 26°41′
В.	0.8437 at 27.6°C on the 12th March 1920.	+ 2°47′ on the 12th March 1920.	0.8706	— 8°37′	0.8708	- 10°10′
C.	0°8472 at 27'6°C on the 12th March 1920.	- 2°82' on the 12th March 1920.	0*8598	— 6°25′	0.8598	- 7°23′
D.	0.8435 at 30°C on the 31st March 1920.	+ 4°54′ on the 31st March 1920-	0.8447	+ 4°34′	C.8482	+ 3°33′

It will be seen that the change is the greatest in the case of sample A and least in the case of sample D. The change seems to be intramolecular and not due to oxidation because samples of steam distilled turpentine kept in two bottles one completely full and the other half full, gave almost identical results.

It was therefore decided to distil off the turpentine with wet steam leaving a hydrated mixture of resin and gum which for convenience was termed 'candy' from its appearance.

An attempt was made to drain out the resin from the candy by melting it in a 80 mesh wire gauze sieve at about  $130^{\circ}$ C. Only about 30% of the resin could be melted out in this way. This resin contained a small quantity of matter insoluble in 90% alcohol.

The method finally adopted enabled a complete separation to be effected. This method originated in the following observation.

It had been noticed while endeavouring to dissolve gum by boiling with water, that any resin present in it readily separated and rose to the surface from which it could be skimmed off. It appeared likely therefore that complete separation could be carried out this way even from the original candy. With the object of effecting the solution of the gum in water if possible, recourse was had to heating a small quantity with water under pressure when it was found that a very clean separation of the gum and resin took place. At the higher temperature obtained in this way the resin becomes very mobile and separates almost completely as a molten mass which solidifies on cooling for the most part as a single lump. The gum forms a flocculent precipitate or mucilage on the bottom of the vessel. The resin can be easily washed free from traces of gum and the gum can be separated from bark chips &c. by washing through a fine sieve.

In the following tables are given the results of these preliminary experiments with small quantities, 424 grams of candy being added to 560 cc of water in a porcelain beaker which was placed in the autoclave the heating being continued for  $1\frac{1}{2}$  hours.

Pressure in the autoclave.	Yield of dry resin calculated on the amount of gum-resin.	Moisture in the hydrated resin	Appearance of the resin-
10 lbs.	47%	21%	Brown and trans- parent.
20),	48 or .o	22%	A little paler than above and transparent.
30 "	ō2%	19%	do
40 "	<b>52</b> %	<b>19</b> %	A little darker than above but transparent

From these results the best conditions would seem reached at 30 lbs pressure.

Preliminary trials being so satisfactory larger quantities were dealt with as follows.

From 25 to 40 lbs gum-oleo-resin were taken in a steam jacketted copper still of 13 gallons capacity into which live steam could be directly introduced by a pipe which projected through the crown of the still into the molten gum-oleo-resin. The steam left the boiler at 30 lbs pressure. The turpentine readily came away and was condensed with the steam. The total quantity distilled amounted to 8 per cent of the crude material.

The 'candy' remaining behind in the still could for the most part be readily melted out through the wide tap at the bottom of the still by the addition of a little steam. A little remained sticking to the sides of the still and could easily be scraped off by hand in removing the still head. In continuous large scale working it would not be necessary to clean out the still every time as the residue from one operation would come away with the next.

The separation of resin from gum was carried out in a large copper container holding about 3 gallons. Some 5 lbs. of candy were treated at a time. The container was placed in a large autoclave and kept at 30 lbs pressure for  $1\frac{1}{2}$  hours. The details of the different experiments were as follows :----

In experiment	(A) 5 lbs of candy were cooked with	
•	3 litres of water, at 30 lbs pressure.	

do

(B) 5 lbs of candy were cooked with 6 litres of water at 30 lbs pressure.

do

(C) 5 lbs of candy were boiled with 6 litres of water and the hydrated resin that rose to the top was skimmed off. It was again boiled with water for some time, the whole operation requiring about an hour.

Experiment.	Yield of dry resin calculated on the amount of gum resin.	Moisture in the hydrated resin.	Matter insoluble in 90% alcohol in th <b>e</b> dry resin	Appearance of the resin.
A	52%	20%	1.6%	Brown a'n d trans- parent
В	54%	36%	1.5%	Practically the same
· C	45 %	34%	26%	A little darker but transparent.

It is evident that the best conditions obtained in experiment B. It would appear necessary to have enough water present to enable complete separation to take place between the gum and the resin and a sufficiently high temperature to ensure that the letter is completely mobile and so does not tend to take up extraneous matter.

The hydrated resin after separation and cooling was removed and washed as indicated above and was dehydrated by heating in an air oven at 130°C. A good deal of frothing takes place at first and constant stirring is necessary. As the dehydration proceeds the melted mass which is originally yellow, opaque and very viscous becomes brown mobile and clear and frothing ceases completely. The melted resin can then be readily filtered through muslin or a fine wire sieve and so separated from the small quantity of finely divided extraneous matter still present.

After removing the resin the residual gum mucilage was allowed to settle and the supernatant liquid carefully decanted off This formed about 60% of the water added and contained about 1.5% of gum in solution which it was not judged worth while to recover. The mucilage after straining off debris &c was carefully evaporated to dryness.

# Description of the products obtained by the cooking process.

For comparison the constants quoted by Pearson and Puran Singh on page 21 of their Memoir are also added.

Dem Tem

### Turpentine

Fractional distillation.	Fowler and Malandkar.	Pearson and Puran Singh.
Fraction passing below 160°C	81.0% (by volume)	50% (by volume)
, do $160^{\circ}C - 180^{\circ}C$ .	14-4%	28.5%
Fraction passing above 180°C. (by difference)	4-6%	22.5%
Specific Gravity	0.8435 at 30°C.	0-8371 at 22°C
		(for bulk fraction after the highe boiling portion were eliminated).
Optical rotation $[\infty]_D$	+4°54'	+ 32°30′
Resin.	(101	the above fraction
Acid value.	51.6	43-7]
Saponification value.	61.3	89.66
Iodine value.	108-1 (Wij's)	97·10 (Hubl)

The resin obtained by the cooking process did not differ appreciably in appearance from that, extracted by a solvern though containing a very small percentage of insoluble matter A sample has been sent to two Bombay firms and their reports

Gum. After drying the gum was obtained as a dark mass which on grinding yielded a grey powder. The yield of dry gum was 23 per cent.

The powder forms a mucilage with water of feeble sticking power. Attempts to reduce the amount of water in the mucilage by filtering were unsuccessful.

The gum obtained by the cooking method was darker than that left after separation of the resin by a solvent and the process appears to cause incipient decomposition of the gum but not so as seriously to alter the composition of the main bulk of the substance.

Attempts at bleaching the gum by chlorine or sulphur dioxide were not very satisfactory.

When mixed with 'blanc fixe' or precipitated barium sulphate in the proportion of about 50% together with colouring matters such as lead chromate, or Prussian Blue fairly good distempers were obtained, and the slight gray tint of the powder was of little moment.

Costs. Assuming that the products are equal in value to those obtained by a solvent extraction process, it remains to compare the question of costs.

The following items are given in Pearsons and Puran Singh's Memoir. Pages 42 and 43.

0	<b>b</b>			
	Cost of dealing with 100 maunds of gum-oleo-resin.	Rs.	А.	Р.
1.	Cost of collecting 100 maunds of crude "drip" at Rs. 5 per maund	500	0	0
2.	Cost of distillation :			
	(1) Loss of 5 per cent. of Benzine (Sp. Gr.=74) at Rs. 2 per gallon on 100 maunds of resin	<b>1</b> 12	0	Û
	(II) Steam distillation, recovery of solvent from both stills per 100 maund, at	50	0	0
	0 - 8 - 0 per maund			
	Total	662	0	0
8.	Labour and supervision at Rs. 0-8-0 per maund per 100 maunds of resin	50	0	0
4.	Over head charges at 2 annas per maund per 100 maunds of resin	12	8	0
5.	Depreciation at 10 per cent. on plant per 100	•.	i -	
	maunds assuming its cost to be Rs. 1,00,000 and its capacity 60,000 maunds of resin per annum	16	12	0
6.	Interest each year at 6 per cent. on Rs. 50,000 working capital per 100 maunds	5	0	0
7.	Packing, insurance, etc., 4 annas per maunds per 100 maunds of resin	25	0	0
8.	Miscellaneous charges	- 8	12	0
Ϋ.	Grand Total	<b>78</b> 0	0	0

#### Receipts.

Turpentine (yielded 8 per cent. Sp. Gr. 84) 77 gallons at Rs. 2-8-0	192	8	0
Resin 55 maunds at Rs. 10 per maund (yield 55 per cent)	550	0	0
Gum (yield 22 per cent) 22 maunds at Rs. 5 per maund	110	0	0
Total	852	8	0

As regards the turpentine steam distillation is common to both processes and consequently the costs may be taken as being identical, though probably the use of low temperature steam will involve less wear and tear of plant than if super heating is employed.

The yield of resin by the cooking process is probably about 1 per cent less than by the solvent process so that at similar prices there would be a loss of Rs. 10 per 100 maunds. The cost due to loss of solvent in the above table is given as Rs 112 so that the net advantage so far of the cooking process is Rs. 102. The question resolves itself into the cost of fuel for heating up the autoclave, for drying the hydrated resin, and evaporating the mucilage. Careful calculation shows the heat required to be about 4/5 of that necessary for steam distillation of the turpentine.

Taking Pearson and Puran Singh's figure for this therefore, the cost of fuel for the cooking process works out at Rs. 40 leaving a balance in favour of the cooking process of Rs. 62 as uming all the other items to be the same.

For the rest the cooking process is much less complicated and less difficult to handle by semi-skilled labour than the solvent process and the capital cost will doubtless be less.

On the whole therefore the cooking process seems the most likely to succeed, in India at any rate, on account of its comparative simplicity. The method may very likely be applicable to the treatment of other gum resins.

The chemistry of the gum of *Boswellia servula* is at present being investigated by the authors.

### Addendum,

At the suggestion of Mr. Pearson to whom a typewritten copy of the foregoing paper was sent for criticism before publication, a general description is appended of the plant and operations which would be required to develope the process on a large scale.

The following items of plant would appear to be necessary :---

(1) A boiler capable of delivering steam say at 80 lbs pressure.

(2) A still provided with closed and open steam coils and necessary condensers.

(3) An autoclave tested to say 40 lbs pressure and provided with a closed steam coil.

(4) An enamelled steam jacketted evaporating pan provided with tipping and stirring gear.

(5) A plain tinned iron evaporating pan.

(6) A small grinding mill or disintegrator.

Taking these items separately :---

(1) A vertical boiler capable of delivering about 400 lbs. steam per hour will be adequate and will probably be found most convenient although the waste heat from the flues cannot be readily utilised.

(2) A still of the type figured in Messrs. Pearson and Puran Singh's Memoir will serve very well for the distillation of the turpentine. To avoid too great dilution of the turpentine with condensed water it will probably be advisable to use both open and closed steam coils, their proper manipulation being a matter of experience.

If possible it will be convenient if the still is placed on an upper platform so that the molten candy can be run direct into the autoclave beneath with consequent economy in heat and labour.

(3) The diagram attached to the present paper shows the character of the autoclave suggested. It may be of tinned iron, heated by a closed coil. An autoclave of the diamensions given in the diagram will serve to heat about 5 cwts. of candy. A grid with a number of hooks is hung in the upper part of the autoclave to facilitate removal of the resin. After heating the candy in the autoclave for about one hour at 20-30 lbs. pressure the whole can be allowed to cool and the mucilage run off from the bottom into any convenient receptacle. The hydrated frozen resin will generally be found at the top and it may be assumed that it will adhere to the hooks and thus be capable of removal in one solid piece, or it may be found more convenient to ladle out the molten material into the evaporating pan, where it can be washed free from any adhering mucilage.

(4) An evaporating pan of the character required can now be obtained from several British firms. It should be shallow rather than deep to facilitate rapid removal of the water. Heating will have to be cautious at first with continued stirring to prevent undue frothing. When the resin has cleared the molten mass can be filtered through a fine meshed sieve or through cotton wool into moulds. Probably shallow tinned iron trays would be best for this purpose.

The mucilage may be concentrated somewhat in a large conical wooden vat in which small extraneous particles of wood and other impurities will settle to the bottom from which they can be removed. The bulk of the mucilage can then be strained through a fine sieve and evaporated to a granular mass in a tinned iron pan over an open fire. The lumps of dried material can be reduced to fine powder in a small disintegrator.

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