

## PHYTOCHEMICAL NOTES.\*

No. 91 Cassia Oils from Leaves and Twigs.<sup>1</sup>

BY K. K. CHEN.

Ever since Schimmel & Company in 1892<sup>2</sup> distilled the several parts of *Cinnamomum Cassia* Blume, the cassia oil situation has been pretty well understood. In connection with the study of Chinese medicinal plants and drugs undertaken in this laboratory, it seemed desirable to learn more about these oils particularly since it became possible to obtain larger amounts of material directly from China. It was for this reason that the experiments here recorded were undertaken. In so far as the leaves and twigs were distilled separately, the results supplement those given by Schimmel & Company.



BALES OF CASSIA LEAVES AND TWIGS.

The leaves were kept compact by bamboo splits (*Bambusa*, Chinese *TsO*), with four bamboo sticks on the four sides. The twigs were similarly bound but required no bamboo sticks. The bales were wrapped in mats made of *Livistoma chinensis* Br. (Chinese, *Boo Pao*) and tied with rattan, a species of *Calamus* (Chinese *Dun*).

*Source and Description of Material.*—Upon the suggestion of Dr. Paul Reinsch, late Ambassador of the United States of America to China, the materials used in this investigation were kindly imported from China by Anderson, Meyer & Co. of New York City. They consisted of five bales of about 100 pounds each: three of leaves and two of twigs, which are shown in the accompanying photograph.

The weights of the individual bales, of their contents, etc., are herewith tabulated.

\* From the Laboratory of Edward Kremers.

<sup>1</sup> Scientific Section, A. Ph. A., Cleveland meeting, 1922.

<sup>2</sup> Bericht von Schimmel & Co., Oct. 1892, p. 12.

Bale No.	Material.	Gross wt., lbs.	Wt. of packing, etc., lbs.	Sample taken out, lbs.	Net wt. for experiment, lbs.
1	Leaves	99	8 <sup>1</sup> / <sub>2</sub>	2	88 <sup>1</sup> / <sub>2</sub>
2	Leaves	102 <sup>1</sup> / <sub>2</sub>	8 <sup>1</sup> / <sub>2</sub>	..	94
3	Twigs	95	5 <sup>1</sup> / <sub>2</sub>	2	87 <sup>1</sup> / <sub>2</sub>
4	Twigs	95 <sup>1</sup> / <sub>2</sub>	5 <sup>1</sup> / <sub>2</sub>	3	87
5	Leaves	104	8 <sup>1</sup> / <sub>2</sub>	..	95 <sup>1</sup> / <sub>2</sub>

*Distillation of the Oil.*—The large still being out of commission, the 60-liter Lentz distilling apparatus had to be used. The leaves being hard they could not be packed well, hence those for the first charge (27 pounds) were cut. The second charge of uncut leaves was 22.7 pounds. Inasmuch as no particular advantage was gained by cutting, this operation was dispensed with. The distillation of a full charge of leaves required two hours and forty minutes, the oil being collected in Florence flasks. The milky aqueous distillate cleared in a day or two with the separation of oily drops. It was cohobated repeatedly, more oil resulting from cohobation than from the original distillation. The volumes that had to be dealt with are herewith tabulated:

	Bale No. 1 of 98 <sup>1</sup> / <sub>2</sub> lbs., gals.	Bale No. 5 of 89 <sup>1</sup> / <sub>4</sub> lbs., gals.	Bale No. 2 of 100 <sup>1</sup> / <sub>4</sub> lbs., gals.
Original distillate.....	50	31	40
After 1st cohobation....	18 <sup>1</sup> / <sub>2</sub>	14	15
After 2nd cohobation....	8 <sup>2</sup> / <sub>3</sub>	6	7 <sup>1</sup> / <sub>2</sub>
After 3rd cohobation....	5	4	5
After 4th cohobation....		5 <sup>1</sup>	
After 5th cohobation....		3	
After 6th cohobation....		2	
After 7th cohobation....		1 <sup>1</sup> / <sub>3</sub>	

The amounts of oil that were separated from the aqueous condensate after each distillation are herewith tabulated:

Original distillate.....	.. <sup>2</sup>	32.7 Gm.=0.08 p. c.	52.5 Gm.=0.12 p. c.
After 1st cohobation....	}	138.8 Gm.=0.34 p. c.	177.7 Gm.=0.39 p. c.
After 2nd cohobation....			
After 3rd cohobation....			
After 4th cohobation....			
After 5th cohobation....			
After 6th cohobation....			
After 7th cohobation....			

<sup>1</sup> From the fourth cohobation on, the aqueous cohobates, also the oily distillates, were mixed for the three bales, hence no separate record for each bale.

<sup>2</sup> Of bale No. 1 the original oil and cohobates Nos. 1, 2 and 3 were not kept separate. The mixed oils amounted to 146.4 grams=0.30 p. c.

The total of 288 lbs. of leaves yielded a total of 658.7 grams of oil or 0.52 p. c. The twigs were cut into lengths of 3 to 4 cm., 54 lbs. constituting a charge, the distillation of which required six and one-half hours. The first charge was distilled with dry steam. The second charge was soaked with water for about twenty-one hours previous to distillation. The former yielded more oil with the original distillate and less from the first three cohobations than the latter. Of about 20 l. of water added to the second charge only 8 l. were recovered as extract after the distillation. Inasmuch as nothing was gained by the soaking, the remaining charges were distilled dry. Both aqueous and oily distillates are again tabulated.

TABLE OF AQUEOUS AND OILY DISTILLATES OF TWIGS (BALES 3 AND 4 = 174<sup>1</sup>/<sub>2</sub> LBS.).

	Water, gals.	Wt. of oil, Gm.
Original distillate.....	64 <sup>1</sup> / <sub>2</sub>	275.2 = 0.34 p. c.
After 1st cohobation.....	26 <sup>1</sup> / <sub>2</sub>	384.9 = 0.52 p. c.
After 2nd cohobation.....	12 <sup>1</sup> / <sub>2</sub>	
After 3rd cohobation.....	6 <sup>1</sup> / <sub>2</sub>	
After 4th cohobation.....	2 <sup>1</sup> / <sub>2</sub>	
After 5th cohobation.....	1 <sup>1</sup> / <sub>2</sub>	
After 6th cohobation.....	1 <sup>1</sup> / <sub>3</sub>	55.7
Total.....		715.8 = 0.90 + p. c.

Whereas 288 lbs. of leaves yielded a total of 658 Gm. or 0.52 p. c. of oil, 174<sup>1</sup>/<sub>2</sub> lbs. of twigs yielded 715.8 Gm. of oil or 0.90 + p. c.

The densities of the leaf oils determined at 22° are herewith tabulated for comparison.

	Original oil.	Oil from Cohobations 1, 2 and 3.	Oil from Cohobations, 4, 5, 6, and 7.
Bale No. 1.....		{ 1.0547 1.0530 }	
Bale No. 5.....	1.0525	1.0540	1.0514
Bale No. 2.....	1.0523	1.0535	
The corresponding data for the twig oils:			
Charge No. 1.....	1.0400	1.0477	1.0473
Charge No. 2.....	1.0420	1.0477	
Charge No. 3.....	1.0393	1.0465	
Charge No. 4.....		.....	

Schimmel & Co.<sup>1</sup> record that the leaves (incl. stalks) and young twigs yielded 0.77 p. c. of an oil the specific gravity of which (apparently at 15°) was 1.055. (Aldehyde content 93 p. c.)

According to the results here recorded, the leaves yielded a total oil of 0.52 p. c. with a density of about 1.054 at 22°, whereas the twigs yielded a total of 0.90 p. c. of oil with a density of about 1.047. It will be seen that the twigs yielded a somewhat larger amount of oil with a slightly lower density than the leaves. The ratio in which leaves and twigs occur on the Cassia shrub does not seem to have been recorded.

## CONTRIBUTIONS TO THE KNOWLEDGE OF FOREST PRODUCTS.

### V. ON NITRO-2, AND AMINO-2 CYMENE.\*

BY W. A. DEMONBREUN AND R. E. KREMERS.

As has been emphasized in the preceding article, the study of cymene and its derivatives forms a unit in the scheme for the development of a more comprehensive policy of forest utilization. This and the succeeding paper are therefore continuations of the effort to shed more light on the chemistry of cymene.

One way of attacking the general problem involved in these researches is to reason by analogy. The idea is not new that cymene can be nitrated, the nitro-compound reduced to an amino-compound, and the latter in turn involved in

\* Based on part of a thesis presented by W. A. D. to the Graduate Faculty of Vanderbilt University in partial fulfilment of the requirements for the Degree of Master of Science, June 1922.