6. Since the previous work gave M. T. D. = 320 mg./Kg. it has been shown that the maximum decrease of toxicity due to amino acid, pure hydrosulphite and centrifugation is 40–60 mg./Kg.

CONTRIBUTION FROM THE
LABORATORIES OF THE
UPJOHN COMPANY,
KALAMAZOO, MICHIGAN.

MISCELLANEOUS CHEMICAL PAPERS.*

4. Fractionation of Turpentine Oil.[1]

BY W. F. SUDRO.

A quantity of approximately five gallons of turpentine was steam-distilled, the distillate being received in approximately liter fractions, sixteen in number. The distillates were immediately placed into bottles filled to the stopper in order to prevent oxidation or other changes. The data on the steam-distillation are given in Table I.

TABLE I.

Steam distillation fraction.	Sp. gr. 20° C. (Westphal).	Refractive index 20° C.	Polarization 23° C.
1	0.862	1.4660	+15.25
2	0.863	1.4675	+14.85
3	0.863	1.4671	+15.1
4	0.864	1.4682	+14.7
5	0.863	1.4680	+14.6
6	0.863	1.4682	+14.5
7	0.864	1.4661	+14.1
8	0.863	1.4668	+13.9
9	0.863	1.4670	+13.8
10	0.864	1.4685	+13.3
11	0.864	1.4684	+13.1
12	0.864	1.4687	+12.4
13	0.865	1.4690	+11.7
14	0.865	1.4690	+10.5
15	0.868	1.4698	+ 9.4
16	0.870	1.4708	+ 7.4

Upon inspection of the table it will be observed that the specific gravity gradually increases from the first fraction to the last, the constants running as one would expect. The polarization constants are also in strict agreement, the angle of rotation gradually diminishing with an increase in the number of the fractions, the only exception noted being either with fraction number two or three.

The indices of refraction show a relative increase with the exception of Fractions 7, 8 and 9. This will be discussed later.

* From the Laboratory of Edward Kremers. Read before Scientific Section, A. Ph. A., Cleveland meeting, 1922.

[1] Part of thesis submitted for the degree of Master of Science at the University of Wisconsin, 1919.

The fractionation here recorded was carred out by Mr. Sudro for the purpose of gaining a better insight into the boiling temperature conditions of a so-called hydrocarbon oil. The results of a carefully performed series of steam and direct distillations of a commercial American turpentine oil may prove of interest to others, hence should be worth recording. The immediate incentive to perform such a "tedious" task was to obtain as pure a pinene fraction as obtainable by this means for chemical experiments that will be reported in another paper.—E. K.

TABLE II.

Steam distillation fraction.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Fraction boiling below 155°																
Percentage of yield...	8.3	2.2	5.0	1.2	1.4	1.8	2.0	2.6	1.6	1.5	2.1	1.0	Negligible amounts distilled			
Specific gravity at 20°	0.861	...	0.859	...	...	...	...	...	...	...	...	...	...	...	...	...
Refractive index at 20°.....	1.4654	1 4648	1.4646	1.4665	1.4663	1.4670	1.4659	1.4659	1.4667	1.4671	1.4670	1.4671	...	...	...	...
Fraction boiling at 155 to 156°																
Percentage of yield...	31.0	28.9	21.7	11.7	8.8	11.5	6.2	6.2	7.6	3.9	2.5	1.6	1.0	Negligible amounts		
Specific gravity at 20°.....	0.861	0.861	0.859	0.862	0.861	0.861	0.861	0.861	0.861	0.862	...	...	...	...	...	...
Refractive index at 20°.....	1.4661	1.4660	1.4659	1.4675	1.4667	1.4671	1.4663	1.4669	1.4672	1.4673	1.4674	1.4671	1.4671	....	...	...
Fraction boiling at 156 to 157°																
Percentage of yield...	41.8	36.6	47.9	25.7	28.7	35.5	37.6	27.7	20.8	21.1	20.8	15.5	4.7	4.5	2.6	1.0
Specific gravity at 20°.....	0.861	0.862	0.860	0.863	0.861	0.861	0.861	0.861	0.862	0.862	0.861	0.861	0.862	0.862	...	...
Refractive index at 20°.....	1.4668	1.4670	1.4670	1.4678	1.4673	1.4674	1.4668	1.4671	1.4675	1.4676	1.4676	1.4677	1.4674	1.4676	1.4680	1.4688
Fraction boiling at 157 to 158°																
Percentage of yield...	9.0	17.0	14.8	36.7	35.8	30.6	32.2	37.3	37.5	40.1	36.9	35.6	24.5	15.2	9.9	1.0
Specific gravity at 20°.....	0.862	0.863	0.861	0.863	0.862	0.862	0.862	0.862	0.862	0.862	0.862	0.863	0.863	0.863	0.863	...
Refractive index at 20°.....	1.4678	1.4678	1.4678	1.4680	1.4677	1.4679	1.4677	1.4677	1.4679	1.4680	1.4680	1.4681	1.4680	1.4680	1.4685	1.4690
Fraction boiling at 158 to 160°																
Percentage of yield...	7.2	11.1	6.8	20.6	19.4	16.6	16.9	19.0	25.2	30.4	33.2	33.6	48.1	52.5	49.6	15.6
Specific gravity at 20°.....	0.864	0.864	0.864	0.864	0.862	0.864	0.864	0.864	0.864	0.864	0.863	0.864	0.863	0.864	0.864	0.867
Refractive index at 20°.....	1.4687	1.4687	1.4687	1.4690	1.4689	1.4690	1.4685	1.4682	1.4685	1.4689	1.4689	1.4686	1.4682	1.4683	1.4687	1.4698
Fraction boiling above 160°																
Percentage of yield...	2.9	4.4	3.9	4.1	6.0	4.2	5.1	7.3	7.4	3.0	4.5	12.8	21.7	27.8	37.9	82.4
Specific gravity at 20°.....	...	...	...	0.880	0.873	0.879	0.876	0.876	0.874	0.877	0.878	0.874	0.871	0.868	0.868	0.873
Refractive index at 20°.....	1.4753	1.4740	1.4765	1.4750	1.4725	1.4742	1.4728	1.4721	1.4723	1.4731	1.4737	1.4719	1.4710	1.4710	1.4716	1.4720

The next step was to distil each of the sixteen steam-distillation fractions over direct flame, fractions being obtained at the following boiling points: up to 155° C., 155 to 156°, 156 to 157°, 157 to 158°, 158 to 160°, and the residue distilling above 160°. The specific gravity and refractive index for each of the separate fractions were obtained. These, together with the percentage of yield, are tabulated in Table II.

The percentage of yield indicates the per cent. by volume of the steam-distillation fractions which distilled at the given temperature. The results obtained are as expected in that the first steam-distillation fractions are richer in the lower than in the higher boiling-point fractions, while those steam-distillation fractions towards the last are richer in the higher than in the lower boiling-point fractions.

A mean is attained, however, with boiling-point fraction 157–158° where it will be noticed that the per cent. of that fraction gradually increases, then remains fairly constant, and finally decreases as the last steam-distillation fractions are reached.

The average specific gravity, the minimum and maximum values for the same, together with the difference between minimum and maximum, are included in Table III.

TABLE III.

Steam distillation fractions.	Average specific gravity.	Minimum specific gravity.	Maximum specific gravity.	Difference between minimum and maximum values.
1	0.862	0.861	0.864	0.003
2	0.863	0.861	0.864	0.003
3	0.861	0.859	0.864	0.005
4	0.866	0.862	0.880	0.018
5	0.864	0.861	0.873	0.012
6	0.865	0.861	0.879	0.018
7	0.865	0.861	0.876	0.015
8	0.865	0.861	0.876	0.015
9	0.865	0.861	0.874	0.013
10	0.865	0.862	0.877	0.015
11	0.866	0.861	0.878	0.017
12	0.865	0.861	0.874	0.013
13	0.865	0.862	0.871	0.009
14	0.864	0.862	0.868	0.006
15	0.865	0.863	0.868	0.005
16	0.870	0.867	0.873	0.006

The same values with reference to the refractive index are given in Table IV.

With reference to Table IV, attention is called to the fact that the average values for the indices of refraction for the steam-distillation Fractions 7, 8 and 9, agree very closely with the constants for the neighboring fractions. This proves beyond a doubt that the variable values given in Table I for these fractions are to be accounted for by the fact that some disturbing factor must have been introduced. In other words, the abnormality was not due to the constituency of the turpentine itself.

Table V includes tabulated data with reference to the specific gravity and refractive index for each of the six boiling-point fractions. The values for the average specific gravity and refractive index are in strict accord with the results which would be expected.

TABLE IV.

Steam distillation fractions.	Average refractive index.	Minimum refractive index.	Maximum refractive index.	Difference between minimum and maximum values.
1	1.4690	1.4654	1.4753	0.0099
2	1.4681	1.4648.	1.4740	0.0092
3	1.4684	1.4646	1.4765	0.0119
4	1.4690	1.4665	1.4750	0.0085
5	1.4687	1.4663	1.4725	0.0062
6	1.4684	1.4670	1.4742	0.0072
7	1.4680	1.4659	1.4728	0.0069
8	1.4680	1.4659	1.4721	0.0062
9	1.4684	1.4667	1.4723	0.0056
10	1.4687	1.4671	1.4731	0.0060
11	1.4688	1.4670	1.4737	0.0060
12	1.4684	1.4671	1.4719	0.0048
13	1.4683	1.4671	1.4710	0.0039
14	1.4687	1.4676	1.4710	0.0039
15	1.4692	1.4680	1.4716	0.0036
16	1.4699	1.4688	1.4720	0.0032

TABLE V.

Boiling-point fractions.	—155°.	155 to 156°.	156 to 157°.	157 to 158°.	158 to 160°.	160°+.
Average sp. gr.	0.860	0.861	0.861	0.862	0.864	0.874
Minimum sp. gr.	0.859	0.859	0.860	0.861	0.862	0.871
Maximum sp. gr.	0.861	0.862	0.863	0.863	0.867	0.880
Diff. betw. min. and max. values.	0.002	0.003	0.003	0.002	0.005	0.009
Average refr. index	1.4662	1.4668	1.4674	1.4680	1.4687	1.4731
Minimum refr. index	1.4646	1.4659	1.4668	1.4677	1.4682	1.4731
Maximum refr. index	1.4671	1.4675	1.4688	1.4690	1.4698	1.4765
Diff. betw. min. and max. values.	0.0025	0.0016	0.0020	0.0013	0.0016	0.0055
Sum total of distillates obt. for given temp.	1.93%	8.94%	23.15%	25.99%	25.49%	14.53%

In Table V the sum total of the distillates obtained for the six boiling-point fractions is also given. Less than 10 per cent. of the entire volume of this particular sample of turpentine consisted of pinene, or the fraction boiling at 155 to 156°; three-fourths distilling between 156 and 160°.

In conclusion, the data submitted show the relation between the several fractions, the results being in close agreement with the theoretical valuation. Slight discrepancies are noted but these are of minor consideration.

CONTRIBUTIONS TO THE KNOWLEDGE OF FOREST PRODUCTS.*

IV. ON CYMENE FROM THE OIL OF MONARDA PUNCTATA L.

BY J. M. JOHNSTON, H. MERRITT, AND R. E. KREMERS.[1]

Introduction.—Among the plants whose volatile oil contains thymol is *Monarda punctata* L. It grows on poor sandy soil in nearly all states east of the Mississippi River. In the north at least it is often found associated with open stands of oak or jack pine. Hence this species of *Labiatae* deserves careful attention in connection

* From the Laboratory for Organic Chemistry, Vanderbilt University.

[1] At present Fellow in Biochemistry at the University of Wisconsin for the National Research Council. This incomplete paper is published as it is, because the work at the Vanderbilt Laboratory has been interrupted.