SCIENTIFIC SECTION

PHYTOCHEMICAL NOTES.*

96. THE VOLATILE OIL OF MYRICA ASPLENIFOLIA ENDL.

BY H. A. BRAUN.†

Gildemesiter records[1] the distillation of three species of *Myrica*, the only genus of the family *Myricaceae* that has thus far yielded a volatile oil, *viz.*, of *Myrica Gale* L., *M. cerifera* L., and of *M. asplenifolia* Endl. The last is recorded by Gray as belonging to a distinct genus, *viz.*, *Comptonia asplenifolia* Ait. Whereas sweet gale oil has been chemically examined by several investigators, bay berry or wax-myrtle oil has been studied with reference to its physical constants only thus far. Sweet-fern oil appears to have been distilled but once, *viz.*, by Schimmel & Co. in 1890,[2] who obtained a yield of 0.08 per cent. They found a specific gravity of 0.926 and add that the oil has a spicy, cinnamon-like odor.

Inasmuch as an opportunity presented itself to distill some of the oil, it seemed highly desirable not to neglect it. About 540 lbs. of fresh material were collected in the vicinity of Pelican Lake and Rhinelander, Wisc., where the plant constituted the undergrowth of the cut-over Jack pine and red-pine forests. The partly dried material yielded upon distillation about 60 cc. of oil or 0.02 per cent as computed for the fresh material. The oil was dark yellow in color and had an odor which somewhat resembled that of new mown hay; $d_{25°} = 0.8945$, hence somewhat lower than that recorded by Schimmel & Co.; $D^{22°} = 1.6629$; $\alpha_{D25°} = -3° 75'$ in a 190-mm. tube. A methoxy determination gave a negative result.

When fractionated, 40 cc. of oil yielded the following fractions of which the densities only were determined.

Fraction.	B. p.	Vol.	$d_{25°}$.
I	−100°	5.0 cc.	0.7346
II	100–150°	4.5 cc.	0.8620
III	150–200°	5.6 cc.	0.8850
IV	200–220°	8.2 cc.	0.9075
V	220°	....	....

Schiff's reagent failed to reveal the presence of even traces of aldehyde in the lowest fraction.

The second fraction, upon saponification, revealed an ester content of 22.5 per cent computed as $C_{10}H_{19}OCOCH_3$. The boiling point, however, is too low for such a high molecular ester.

The third fraction (150–200°) revealed a similar ester content. When treated with semicarbazide hydrochloride it yielded a crystalline compound, too small in amount for recrystallization, that melted at 249°. With Schiff's reagent this fraction gave a decided test, thus indicating that the semicarbazone may be that of an aldehyde present in this fraction.

Fraction 200° to 220°, upon saponification, revealed an ester content of 28.28 per cent computed as $C_{10}H_{19}OCOCH_3$. Acetylation revealed a total alcohol con-

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[1] *Die aetherischen Oele*, Bd. II, p. 327 *et seq.* [2] *Bericht S. & Co.*, p. 50 (Oct. 1890).

tent of 45.74 per cent computed as $C_{10}H_{19}OH$. Hence, free alcohol is present to the extent of 17.46 per cent. Treatment with phenyl isocyanate yielded diphenyl urea, m. p. 230°, but no urethane.

It is apparent that a much larger amount of oil will be necessary to reveal its chemical composition.

STUDIES IN THE GENUS MENTHA, XI.

THE OIL OF MENTHA CANADENSIS, L.

BY H. A. BRAUN.*

The first oil to be distilled was reported by Schimmel & Company[1] in 1893 from material collected at Passiac, N. J. In 1898, Florence M. Gage[2] distilled an oil from material collected in the neighborhood of Madison. A larger quantity of of oil, distilled from material collected by O. A. Beath in Wyoming, was examined by R. E. Kremers[3] in 1923. During the summer of 1925 the opportunity presented itself to obtain material from the northern part of Wisconsin. About 450 pounds of fresh flowering material were collected between August 3rd and August 24th at Crandon, Wisconsin, along the banks of Swamp Creek, the outlet of Stone Lake, and in the marshes which border this stream. This yielded 170 pounds of dried herb which, in turn, yielded 936 grams of oil or 1.16 per cent.

Schimmel & Company[1] report a yield of 1.23 per cent from the dried herb. Miss Gage[2] reports 1.25 per cent from the dried herb. R. E. Kremers[3] reports a yield of 1.80 per cent of primary oil and 0.36 per cent of cohobated oil, making a total of 2.16 per cent.

The oil was of a greenish yellow color and possessed a penetrating minty odor. The physical and chemical constants are herewith tabulated with those of previous investigators for the sake of ready comparison.

	H. A. B.	R. E. K.	F. M. G.
$d_{25°}$	0.8974	0.931	−0.927 to 0.935
$\alpha_{D^{25}}$ 10 cm. tube	+32° 24′	+18.75°	+16° 11′ to 20° 32′
Phenol assay	none	none	traces of thymol or carvacrol
Ester value	22.78	11.2	
Per cent ester ($C_{10}H_{19}OCOCH_3$)	8.06 per cent	4.0 per cent	
Ester value after acetylation	32.00 per cent	33.6	
Per cent total alc.	15.69 per cent	9.6 per cent	
Per cent free alc.	7.90 per cent	6.5 per cent	
Pulegone assay (neutral sulphite)	20.00 per cent	90 per cent	
Pulegone assay (acid sulphite)	18.00 per cent		

Though the oil agrees better with the other Wisconsin oil than with the Wyoming oil, it differs appreciably even from the former. Five hundred and thirty-six grams of oil, when shaken with a 5 per cent solution of potassium hydroxide yielded no more than a few drops of oil on acidification. Flueckiger's test for thymol and carvacrol gave a negative result.

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[1] *Bericht S. & Co.*, p. 45 (Oct. 1893).

[2] *Pharm. Rev.*, 16, p. 422.

[3] JOUR. A. PH. A., Vol. XIV, p. 32.