Fraction.	B. p. 11.	d_{25}.	n_{25}.
9	110–20	1.928	1.4800
10	120–30	0.951	1.495
11	130–40	.....	1.500
12	140+	Residue	.....

Menthol.—The chief fractions, notably those boiling between 97 and 104° at 11 mm., crystallized on being exposed over night to temperatures below 0° C. By allowing the oils to freeze in separatory funnels or in percolators, the liquid portions could be drained with considerable success. By melting the crude crystals, refreezing, and draining again, pure menthol was isolated in some quantity. In a more severe winter than that of 1920–21, this method of isolation and purification should prove to be quite efficient.

Menthone.—A test portion of the fractions from which menthol had been frozen out was treated with semicarbazide solution. The semicarbazone thus obtained was purified by digestion with hot dilute alcohol; m. p. was 185°. Menthone semicarbazone melts at 184°; therefore menthone was present.

Methyl-1 Cyclohexanone-3.—The constants recorded for methyl-1 cyclohexanone-3 are: b. p. = 169° C.; $d_{21} = 0.915$; $n_D = 1.4430$. Those of Fraction 1 are in good agreement with these values. A preliminary trial yielded a semicarbazone which had a melting point of 180° after recrystallization, namely, that of methyl-1 cyclohexanone-3 semicarbazone. Accordingly the presence of this compound is indicated.

SUMMARY.

The results of this investigation of the composition of the cohobated oils of American and Japanese peppermints established the following:

1. The cohobated oil of American peppermint is composed largely of menthol and menthone; menthylesters and methyl-1 cyclohexanone-3 are lesser constituents.

2. The cohobated oil of Japanese peppermint contained no isolable quantity of menthol, which was contrary to expectation, but on fractionation yielded almost exclusively the pulegone fraction.

3. No explanation is offered for the fact that no alcoholic constituent was isolated from the Japmint oil after chemical assay had indicated the presence of 20 percent of alcohol calculated as menthol, unless the usual assay methods are not applicable to oils containing a high percent of pulegone.

A RAPID ASSAY METHOD FOR THE DETERMINATION OF ASCARIDOLE IN OIL OF CHENOPODIUM.*

BY E. K. NELSON.

On account of the increasing use of oil of chenopodium as a treatment for hookworm in man and animals, it is desirable to have a quick, reliable method for the estimation of ascaridole, which is undoubtedly the active ingredient.

At present the oil is judged by its physical constants, and the Pharmacopoeia provides that it should have a specific gravity at 25° of 0.955 to 0.980, an optical rotation of —4° to —10°, and be soluble in 8 volumes of 70% alcohol.

* Contribution from the Essential Oils Laboratory, Drug Division, Bureau of Chemistry, U. S. Department of Agriculture. Presented to Scientific Section A. PH. A., New Orleans meeting, 1921.

Since ascaridole is quite unstable under certain conditions, the oil is very liable to deteriorate on keeping especially if precautions are not taken to keep cool and exclude light.[1] Quite a large proportion of the ascaridole might become thus altered without changing the physical constants so that they would fall without the limits prescribed by the Pharmacopoeia.

The estimation of ascaridole in the oil was accomplished by Schimmel and Company,[2] by fractional distillation, and is stated by them to be from 62 to 65% in normal oil of specific gravity 0.9708, from 45 to 50% in light oil, specific gravity 0.9426, and 65 to 70% in an oil distilled by themselves.

The residue left on vacuum distillation[3] seems to be of some value in indicating to what extent the ascaridole has become altered, but as the results depend on the degree of vacuum employed and the temperature at which the distillation is carried on, they can only be an approximate measure of the non-volatile, resinous or polymerized material.

The method here proposed depends on an observation of Flu, DeLangen and Weehuizen[4] that ascaridole is soluble in a mixture of 60 parts by volume of glacial acetic acid with 40 parts of water.

In order to determine the action of the reagent on the terpenes of the oil, 10 Cc. of chenopodium terpenes were shaken with 60% acetic acid in a cassia flask and found to be insoluble. 10 Cc. of ascaridole, similarly treated, dissolved to the extent of 98% and the residue remaining was a viscous oil which was probably altered or polymerized ascaridole.

Accordingly all that is necessary to rapidly estimate ascaridole in oil of chenopodium is to agitate 10 Cc. thoroughly in a cassia flask, the neck of which holds 10 Cc. graduated in tenths, with 60% acetic acid.

The flask is then filled to the mark with 60% acetic acid and allowed to settle, or carefully centrifuged. The volume of undissolved oil deducted from 10 and multiplied by 10, gives the volume percentage of ascaridole in the sample.

The following are some results obtained by this method:—

Sample No.	$d\frac{25}{25}$.	Rotation.	Solubility.	Per cent ascaridole. By distn.	By acetic acid.
1.....	0.9564	—6.5°	O.K.	64	70
2.....	0.9640	—5.0°	O.K.	..	70
3.....	0.9564	—5.65°	O.K.	..	64
4.....	0.9704	.....	....	..	73
5.....	0.9325	.....	....	..	48

A REPORT ON THE ZAMIA STARCH SITUATION.*

BY JOSEPH F. CLEVENGER.

One region in Florida where the Zamia plant, *Zamia floridana* DC., grows was visited and a number of the plants were dug up and examined. In addition, the only mill manufacturing starch from the plant was visited. The following information was collected:

[1] Nelson, *Cir. Bur. Chem.*, No. 109, Jan. 1913.

[2] Schimmel and Company, "Semi-Ann. Report," April 1908.

[3] Nelson, *Jour. Am. Chem. Soc.*, 42, p. 1204.

[4] Mededeel. u. het. geneesk. lab. te Weltervreden, 3rd ser. A, 1919, pp. 1–28.

* Presented at the sixty-ninth annual meeting of the American Pharmaceutical Association, New Orleans, La., September 6–11, 1921.