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THE VOLATILE OIL OF COMPTONIA ASPLENIFOLIA.*

BY ROMULUS DE NICOLA AND E. V. LYNN.¹

The so-called sweet fern, which grows prolifically in many regions of Massachusetts, is *Comptonia asplenifolia*, *Aiton (Myricaceæ)*. It is found in sterile soil from Nova Scotia to Saskatchewan and southward to North Carolina and Tennessee. All parts of the plant contain a resinous substance, but the characteristic odor is apparently due to the volatile oil found chiefly in the leaves.

In 1890 Schimmel & Co. obtained² 0.08 per cent of an oil with a specific gravity of 0.926. Later Braun distilled³ 0.02 per cent of an oil with a specific gravity of 0.8945 and a slight levorotation. He fractionated the oil and reported the presence of aldehydes and esters but identified no specific compounds.

Our material was gathered in the summer of 1938 in Massachusetts and New Hampshire and was identified by Dr. Youngken. Upon drying in the air it lost a total of 63.14 per cent. Distillation of 130 pounds of the fresh leaves and stems gave a total of 31.5 Gm. of oil, a yield of 0.054 per cent, or 0.14 per cent on the dry basis. It was yellowish brown in color, with a cinnamon-like odor, a specific gravity of 0.9154 at $20^{\circ}/20^{\circ}$ C., an index of refraction of 1.4870 at 20° C. and a congealing point of about 5° C. The acid value was 14.04, ester value 13.30, and saponification value after acetylization 58.66. The color prevented determination of optical rotation.

The acids, extracted by sodium carbonate, were distilled and titrated (Du Claux method) to give values of 6.6, 6.6 and 7.5. Theoretical for formic, acetic and propionic are 4, 7 and 12, respectively. From the acid mixture could be extracted by ether a small amount of substance which was precipitated with silver nitrate. The ignited precipitates left residues of 43.21 and 33.70 per cent of silver, indicating acids with molecular weights of 142.9 and 213.4, respectively.

The phenolic fraction, extracted by alkali, amounted to 0.24 per cent. From it no bromide or phenylurethane could be obtained, and other reactions indicated that the substance consisted chiefly of lactone.

The original oil gave no reaction with Schiff's reagent or with semicarbazide hydrochloride which would indicate that the oil contains no aldehydes nor ketones.

Distillation at 18 mm. pressure yielded 20 per cent below 70° C., probably terpenes. No crystalline nitrosochloride could be obtained, although the mixture was cooled greatly.

The fraction between 70° and 150° C. amounted to 35 per cent. It was found to contain cincol by converting to the iodol compound melting at 110° C. Borneol could not be identified.

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² Report October, 1890, page 61.

³ JOUR. A. PH. A., 15, 336-337 (1926).

The oil boiling above 150° C. constituted about half of the total. The presence of caryophyllene was indicated by odor and physical constants, but we were unable to isolate any derivatives. The characteristic odor of the oil seems to reside in this upper fraction.

SUMMARY.

1. The fresh leaves gave 0.05 per cent of yellowish brown oil whose constants are recorded.

2. The oil contains about 20 per cent of terpenes, a considerable amount of cineol and smaller quantities of esters and alcohols, but no aldehydes nor ketones. Acetic acid and small amounts of unidentified acids and lactones are also present.

3. The chief constituents are high boiling and may include caryophyllene.

A COMPARATIVE STUDY OF THE THREE RECOGNIZED ASSAYS FOR OIL OF CHENOPODIUM.*

BY WILLIAM F. REINDOLLAR.¹

Chenopodium or American Wormseed Oil has long been employed as an anthelmintic against round worms in man and stock animals, and more recently has come into use as an agent in the treatment of hookworm.

The principal constituents of the oil are ascaridole, an organic peroxide occurring to the extent of 60–80 per cent, and a hydrocarbon fraction containing cymene, limonene, sylvestrene, phellandrene, etc. Of these components it has been shown that ascaridole alone exhibits anthelmintic action (1), hence should be used as the basis for evaluating the oil. As ascaridole readily reverts to an inactive form, ascaridole glycol anhydride when distilled with steam, as in its removal from the plant, it is highly desirable to employ a method which distinguishes between these two substances.

Assay processes for wormseed oil are given in the Official and Tentative Methods of the Association of Official Agricultural Chemists (2), and in the British (3) and United States Pharmacopœias (4). The first two procedures determine ascaridole, while the last named measures the acetic-acid soluble fraction of the oil. The A. O. A. C. method is a modification of one first proposed by Humphrey Paget (5), and which involves the reduction of ascaridole by titanous chloride; the British Pharmacopœia has adopted the Cocking and Hymas (6) procedure based on the liberation of iodine from potassium iodide by the organic peroxide ascaridole; while the U. S. P. employs the Nelson (7) method which depends on the differential solubility of the organic peroxide and hydrocarbon fractions in 60 per cent acetic acid.

Although Broughton and Weiland (8) have demonstrated the merits and limitations of the A. O. A. C. procedure and pointed out its superiority over the method official in the U. S. P. X (9), it was not adopted by either the British or

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¹ From the Laboratories of the Bureau of Chemistry of the State of Maryland Department of Health.