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## A NOTE ON THE VOLATILE OIL OF ILLICIUM PARVIFLORUM MICHX.*

BY P. A. FOOTE.[1]

The phytochemist in Florida has at his door many plants which should be investigated. If he fails to look for them the chances are that some will be brought to him. The writer has five new volatile oils awaiting investigation. These were brought to him by a farmer looking for commercial possibilities. Several years ago our attention was called to the large amount of oil in the leaves of *Illicium parviflorum Michx.* (Fam. *Magnoliaceæ*). This is a shrub 1–2 m. tall and according to Small (1) the habitat is in low woods and swamps in the coastal plain from Florida to Georgia. The appearance of tiny oil cells on the under side of the leaves is very pronounced. When crushed a strong sassafras odor is obtained. This is due to the high safrol content which we found to be higher than in any other oil yielding this compound. A search of the literature indicated that this oil has never been reported on.

### EXPERIMENTAL.

The leaves were collected in Gainesville in October 1933. Upon steam distillation, 2.6 Kg. gave 12.1 Gm. of a colorless oil heavier than water. It stood in diffused light for three years, during which time it turned slightly yellow. A determination of the acid and ester values indi-

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cated but traces of them. Due to the small amount of the oil on hand and its pronounced sassafras odor it was decided to forego the determination of any more chemical and physical constants and utilize the entire amount for the determination of the chief constituent.

*Separation of Safrol.*—8.7 Gm. of the oil were chilled in a mixture of ice and salt to −11° C. No solidification took place even though the melting point of safrol is +8° C. Neither would a commercial sample solidify at this temperature which bore out statements in the literature that a temperature of −12° C. is necessary to solidify safrol. The oil was transferred to a centrifuge test-tube and frozen by dry ice. It was then placed in an ice-bath at 0° C. and kept there for one hour, after which it was briefly centrifuged at 1500 r. p. m. The separated liquid was poured off. It amounted to but five drops and had an odor remindful of nutmeg. An effort to prepare a solid bromine derivative of it failed.

*Identification of Safrol.*—The tube of crystals was immersed in a water-bath held at 11° C. They slowly melted. To determine the melting point a corrected Anschutz thermometer was immersed in the liquid and frozen there by dry ice. The tube was transferred to a water-bath held at 13° C. and the melting point observed to be 7–8° C. This was checked twice. The melting point of safrol was recorded by Eykman (2) as being 8° C. Refractive index at 20° C. was found to be 1.5340. That reported by Schimmel and Co. (3) for safrol is 1.5377 at 20° C. Using a 2.5 cc. weighing tube $d_{\frac{15}{15}}$ was 1.089 as compared to Eykman (2) 1.0960. Safrol was further identified by oxidation with an alkaline potassium permanganate solution which gave the piperonylic acid described by Fittig and Mielch (4). This oxidation must be carefully controlled because of the variety of products obtainable. Repeated trials were first made on commercial safrol. The following procedure was then adopted. Disperse 4 cc. of the sample in 240 cc. of a 1% sodium hydroxide solution contained in an 800-cc. flask. To this is slowly added with agitation 200 cc. of 5% potassium permanganate solution. Heat on a water-bath for one hour. Filter hot and cool. Acidify the filtrate with sulfuric acid. The precipitated piperonylic acid is filtered off, washed with water and recrystallized from hot alcohol. It melts at 228° C.

SUMMARY.

1. The volatile oil of *Illicium parviflorum Michx.* is largely safrol estimated at more than 90%.

2. This oil has the highest safrol content of any volatile oil yet reported.

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# NOTES ON THE STABILITIES OF ATROPINE AND HYOSCYAMINE IN SOLUTION.*

BY H. H. FRICKE[1] AND K. L. KAUFMAN.[2]

The variable amounts of alkaloids obtained in the assays of the Solanaceæ drugs have been the cause of much study of the various processes. Several factors may explain the variations in the amount of alkaloids isolated. One may be the instability of atropine and hyoscyamine when heated in the various solvents. Another factor is the presence of amines and ammonia in the drugs.

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