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## Note

### Gas-liquid chromatography of some tropolone-TMS ethers

E. L. JOHNSON and A. J. CSERJESI

Department of the Environment, Canadian Forestry Service, Western Forest Products Laboratory, Vancouver, B.C. V6T 1X2 (Canada)

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Some tropolones are among the most powerful natural fungicides, their toxicity varying greatly even between isomers. The thujaplicins are not exceptions to the rule<sup>1</sup>. Early determinations of thujaplicins in western red cedar (*Thuja plicata* Donn) was carried out by a colorimetric assay<sup>2</sup>. This, however, gave no separation of isomers. Chromatography on treated paper<sup>3,4</sup> yields separation, but the method is tedious. Gas-liquid chromatography (GLC), however, has resulted in an easy and adequate separation of the  $\beta$ - and  $\gamma$ -thujaplicin-trimethylsilyl (TMS) ethers, which can be used for quantitative determinations. The conditions for it are presented in this note.

The gas chromatograph was equipped with flame ionization detector and a 3-mm  $\times$  150-cm glass column packed with 5% SE-30 on Aeropak-30. The column temperature was 125° (isothermal), while the injector and detector temperatures were 250°. The nitrogen carrier gas flow-rate was 30 ml/min. Derivatives of tropolones were prepared by adding 500  $\mu$ l of BSA (N,N-bis (trimethylsilyl) acetamide) from Aldrich to 500  $\mu$ g of each of the thujaplicins in small test tubes. The tubes were immediately sealed and placed in a water-bath at about 70° for 30 min. After cooling, 1- to 2- $\mu$ g injections were made, producing symmetrical, adequately separated peaks on GLC. Elution times were:  $\beta$ -thujaplicin-TMS ether at 8.0 min and  $\gamma$ -thujaplicin-TMS ether at 9.2 min. A greater elution time and a higher column temperature were required for nootkatin-TMS ether. Nootkatin is an isopentenyl tropolone present in the heartwood of *Chamaecyparis nootkatensis* (D. Don) Spach and other wood species.

## REFERENCES

- 1 J. W. Roff and E. I. Whittaker, *Can. J. Bot.*, 37 (1959) 1132.
- 2 H. MacLean and J. A. F. Gardner, *Anal. Chem.*, 28 (1956) 509.
- 3 E. Zavarin and A. B. Anderson, *J. Org. Chem.*, 21 (1956) 332.
- 4 C. A. Wachmeister and B. Wickberg, *Acta Chem. Scand.*, 12 (1958) 1335.