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Note

Liquid chromatographic determination of submicrogram amounts of ipsenol and ipsdienol, pheromone components of *Ips paraconfusus* Lanier

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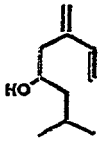
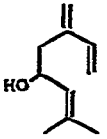
Ipsenol (I, 2-methyl-6-methylene-7-octen-4-ol) and ipsdienol (II, 2-methyl-6-methylene-2, 7-octadien-4-ol) are components of the aggregation pheromone of the bark beetle *Ips paraconfusus* Lanier[1]. These pheromone components were dispersed into the forest environment from slow release devices in bark beetle control experiments.



(I)



(II)

This paper describes an analytical method developed to assess the quantity and quality of ipsenol and ipsdienol remaining after their elution in field experiments.

Gas chromatography was used previously to analyze *Ips* pheromone components[2,3]; however, certain *Ips* pheromone degradation products, such as their polymers, are not volatile and will not readily elute from a gas chromatographic column. I found that a liquid chromatograph with a variable-wavelength ultraviolet detector can be used to determine purity of the pheromone components.

EXPERIMENTAL*

Apparatus

Ultraviolet spectra were determined in acetonitrile in a Unicam SP800 spectrophotometer. Chromatograms were run with a Waters Assoc. (Milford, MA, U.S.A.) Series ALC-200 chromatograph fitted with a U6K injector, a Tracor Model 970 detector, and a Linear Model 555, 10-in. recorder.

* Trade names and commercial products or enterprises are mentioned solely for information. No endorsement by the U.S. Department of Agriculture is implied.

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Column. A Waters Assoc. 30 cm × 3.9 mm I.D. column packed with 10-μm Porasil was used.

Mobile phase. Acetonitrile (Burdick & Jackson Labs., Muskegon, MI, U.S.A.) was the mobile phase.

Reagents

Racemic ipsenol and ipsdienol were from the Chemical Samples Co. (Columbus, OH, U.S.A.) and were freshly distilled under reduced pressure.

Procedure

The pheromone components were dissolved in acetonitrile before injection. The solvent was degassed under vacuum before using. Calibration of standard curves were determined by triplicate injections of known quantities of ipsenol and ipsdienol at several absorption units full scale (a.u.f.s.).

RESULTS AND DISCUSSION

A typical chromatogram of ipsdienol exposed to the forest environment is shown in Fig. 1. Exposed ipsenol except for an earlier elution time has almost an identical chromatogram. Analyses by this method showed degradation of ipsenol from 5 to 10% and of ipsdienol from 1 to 2% when these pheromones components were exposed to the environment. There was also no cross contamination of one

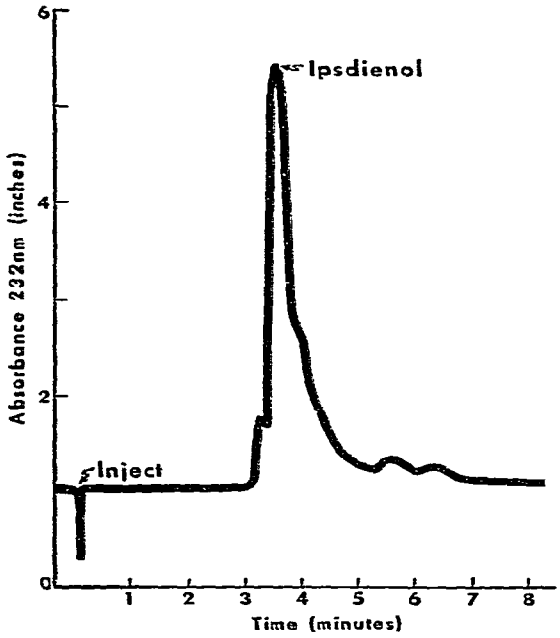

Fig. 1. Typical chromatogram of ipsdienol after exposure to forest environment. Separation on a μPorasil column (30 cm × 3.9 mm I.D.) at ambient temperature with acetonitrile at 1.1 ml/min.

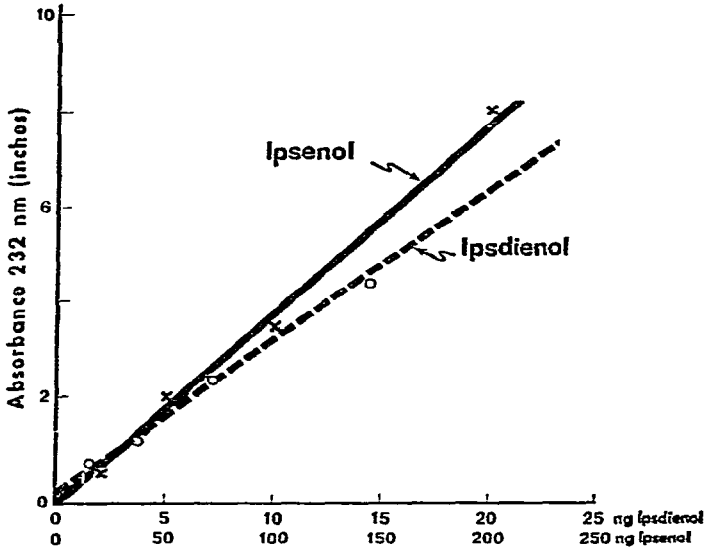

Fig. 2. Peak height *vs.* quantity of pheromone at 232 nm on 10 mV, 10-in. recorder. Ipsenol: 0.04 a.u.f.s.; ipsdienol: 0.01 a.u.f.s.

component into the other in our slow release devices. The two components can be detected at the submicrogram level (0.01–0.005 μg) if necessary.

The λ_{max} of both ipsenol and ipsdienol in acetonitrile is at 225 nm with log ε_{max} at 3.97 and 4.38, respectively. Almost no absorption occurs at 254 nm. The UV absorbance was monitored at 232 nm as a compromise between solvent impurities and pheromone absorbancy. With an eluent flow-rate of 1.1 ml/min and an average pump pressure of 300 p.s.i., the retention time was 3.3 min for ipsenol and 3.6 min for ipsdienol. Calibration curves show that the minimal detectable amount of ipsenol at 0.01 a.u.f.s. is about 5–19 ng, and for ipsdienol it is about 2–4 ng. At the ranges shown, the detector response is linear (Fig. 2). These sensitivities may prove useful in measuring release rates of the pheromones in slow release devices and residual amounts in the environment.

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