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Note

Preparative high-performance liquid chromatography of (2E)- and (2Z)-farnesol

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Cembrene A (Fig. 1) is one of the most interesting biologically active compounds of insect origin. It was originally found in *Nasutitermes exitiosus* termites^{1,2},

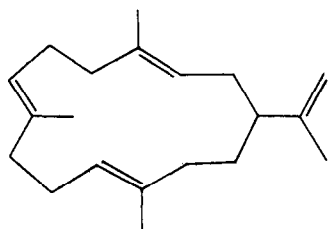


Fig. 1. Structure of cembrene A.

where it has the function of a "trail" pheromone, whereas in *Cubitermes umbratus* soldiers³ it serves as a defensive substance. Pure (2E)- and (2Z)-farnesols (Figs. 2 and 3) are used for the synthesis of cembrene A and its analogues by the procedure of Kato *et al.*⁴. Each isomer is extracted from the natural mixture, in quantities of at least 50 g, by preparative high-performance liquid chromatography (HPLC) because this is the most effective and the fastest method. The conditions for separation of the *E* and *Z* isomers from the natural mixture were optimized on an analytical column and then used for the preparative work. The purity of the fractions was tested by gas-liquid chromatography (GLC). When the separation was not satisfactory, the evaporated fractions were combined and chromatographed again.

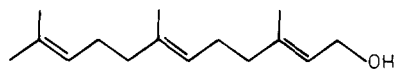


Fig. 2. Structure of (2E)-farnesol.

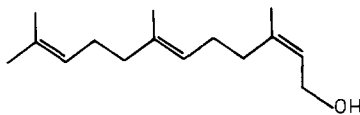


Fig. 3. Structure of (2Z)-farnesol.

EXPERIMENTAL

Apparatus

Analytical chromatography. The chromatographic system consisted of a Milton-Roy Mini Pump (LDC, U.S.A.), a sampling valve (Knauer, F.R.G.) and a refractometric detector RIDK 101 (LP, Praha, Czechoslovakia). A stainless-steel column (250 × 4 mm I.D.) was packed with 5 μm Separon Si (LP). The mobile phase was a light petroleum–ethyl acetate mixture.

Preparative chromatography. The chromatographic system consisted of a Prep-100 chromatograph (Jobin-Yvon, France) and a refractometric detector RIDK 100 (LP). A column (1000 × 80 mm I.D.) was packed with 15–30 μm silica gel prepared according to Pitra and Štěrba⁵.

Gas-liquid chromatography. We used gas chromatograph Chrom 4 (LP) and a column (2500 × 3 mm I.D.) packed with 3% OV-17 on Gas Chrom Q (100–120 mesh). The oven temperature was 150°C, the injector temperature 200°C.

RESULTS AND DISCUSSION

The resolution $R_s = 2$ (Fig. 4) was achieved on an analytical column using light petroleum (b.p. 40–60°C)–ethyl acetate (90:10) as the mobile phase. For pre-

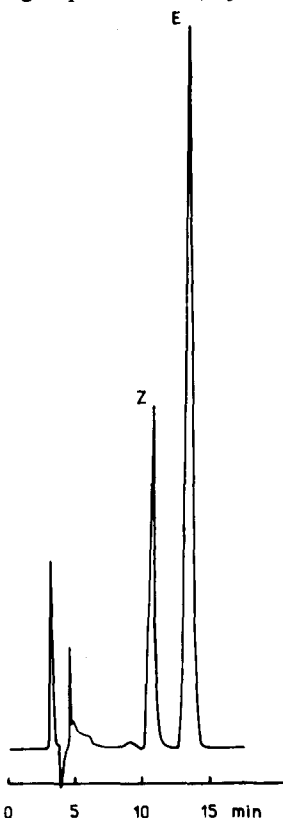


Fig. 4. Analytical separation of farnesol isomers. Column, Separon Si; mobile phase, light petroleum (b.p. 40–60°C–10% ethyl acetate; flow-rate, 1.0 ml/min; RI detection).

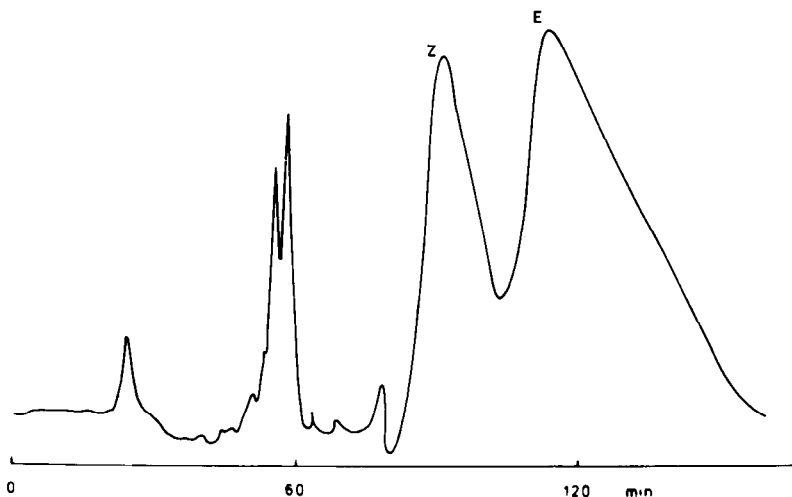


Fig. 5. Preparative separation of farnesol isomers. Column silica gel according to Pitra and Štěrba⁵; mobile phase, light petroleum (b.p. 40–60°C)–ethyl acetate (93:7); flow-rate 110 ml/min; RI detection.

parative chromatography, a mobile phase consisting of light petroleum with 7% ethyl acetate was used at a flow-rate of 110 ml/min. From the point of view of the effectiveness of separation, it was more convenient to overload the column even if the separation of both isomers was not complete. Figure 5 shows a chromatogram of the separation of 40 g of a mixture of the two farnesol isomers on the column (1000 × 80 mm I.D.) (1.5 kg of sorbent). In this way, 8 g of (2Z)- and 24 g of (2E)-farnesol were obtained after 3 h. Column fractions (1 l) were tested by GLC. Insufficiently separated fractions were chromatographed again. In this way, 133 g of (2E)- and 66 g of (2Z)-farnesol were obtained from 12 injections in a relatively short time and using a very cheap sorbent.

REFERENCES

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