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## Note

## Paper chromatography of the essential oils occurring in the genus Stachys

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Some time ago I obtained good separations of mixtures of polycyclic hydrocarbons using a very simple chromatographic system, namely Whatman No. 4 paper impregnated with paraffin oil (10% in petroleum ether) with ascending development using methanol as the mobile phase<sup>1</sup>.

I have also obtained good separations in the analysis of the essential oils of the genus Stachys. These oils have two main constituents which were previously separated by column chromatography<sup>2</sup> and which fluoresce under UV light. They are: 1, a sesquiterpenic enol, stachynone or sesquinol, 1-isopropyl-4,7-dimethylbi-

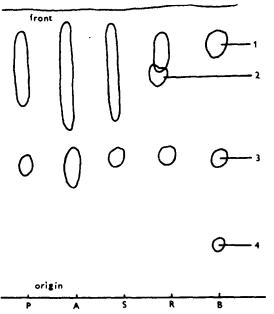


Fig. 1. The paper chromatographic patterns of the essential oils from *Stachys palustris* (P), *S. annua* (A), *S. silvatica* (S), *S. recta* (R), and *S. betonica* (B). Spots; 1 = stachynone, faint blue fluorescence with touch of yellow; 2 = quenched spot of an unidentified compound; 3 = stachynene, blue fluorescence; 4 = unidentified compound, greenish fluorescence.

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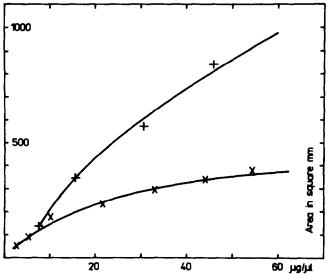


Fig. 2. The planimetric curves of stachynone (upper) and of stachynene (lower).

cyclo [4.4.0]10-decaene-6-one, m.p. 54–55°C,  $[\alpha]_{D^2}^{22} = -2.45$  in tetrachloro methane,  $\lambda_{\text{max.}} = 210$  and 264 nm,  $\lambda_{\text{exc.}} = 360$  nm,  $\lambda_{\text{em.}} = 395$  and 460 nm (the major peak), relative intensity 4.0,  $\lambda_{\text{exc.}} = 520$  nm,  $\lambda_{\text{em.}} = 545$  nm, relative intensity 2.2,  $R_F$  0.93 (an oblong spot); 2, its parent bicyclic sesquiterpene of the cadalene type, 1-isopropyl-4,7-dimethylbicyclo[4.4.0]6,10-decadiene,  $d_4^{21}$  0.8762,  $n_D^{21}$  1.4794,  $\lambda_{\text{max.}} = 227$ , 245, 277, 284 and 330 nm,  $\lambda_{\text{exc.}} = 360$  nm,  $\lambda_{\text{em.}} = 450$  nm, relative intensity 39.8,  $R_F$ 0.40.

During the column chromatography a waxy material was also isolated from the oil of *Stachys annua* which had  $R_F$  values of 0.60 and 0.36 (m.p. 34 and 127°C) and which may give rise to a grey fluorescent background on the chromatograms.

After development on the paper (Whatman No. 3 or 17) stachynone can be isolated from the paraffin used as stationary phase by elution with diethyl ether on a silica column and stachynene with pentane on a Florisil column. The chromatograms were usually run in glass cylinders (diameter 15 cm, height 30 cm) with a ground-glass lid. Typical chromatograms are shown in Fig. 1. A semi-quantitative evaluation by spot area measurement is possible. The relationship for a typical chromatogram is shown in Fig. 2.

## REFERENCES

- 1 E. Malý, J. Chromatogr., 40 (1969) 190-191.
- 2 E. Malý, Miltitzer Ber., (1982) 27-29.