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2,4,5-TRIMETHOXYSTYRENE FROM *PACHYPODANTHIUM STAUDTII**

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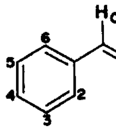
Key Word Index—*Pachypodanthium staudtii*; Annonaceae; 2,4,5-trimethoxystyrene; synthesis.

Plant. Pachypodanthium staudtii Eng. *et* Diels. *Voucher specimen.* Enti 1296, deposited at the herbarium of the Royal Botanic Garden, Edinburgh. *Source.* Rain forest in the West Neung Forest Reserve, near Dompin, Ghana. *Uses.* In native medicine [1]. *Previous work.* Traces of alkaloid in leaf and bark [1]. *Plant parts examined.* Root and stem barks, wood and leaves.

Present work. Ground root bark (500 g) was extracted in a Soxhlet with petrol (40–60°) Concn of the petrol extract gave a yellow deposit which on recrystallisation from aq. Me_2CO gave white plates (2·16 g) mp 60°. This compound, which was optically inactive, gave the following spectral data. UV λ_{max}^{EtOH} nm (log ϵ): 258 (4·09), 264*sh* (4·05), 314 (3·83) unchanged on addition of acid or alkali. IR ν_{max} cm^{-1} (KCl): 1610, 1510, 1460, 1335, 1310, 1265, 1210, 1040, 900, 860, 840. MS: Found M^+ 194·0942, $C_{11}H_{14}O_3$ requires 194·0943, with major fragments at *m/e* 194 (100%), 179 (M^+-Me), 151 (M^+-Me,–CO). PMR ($CDCl_3$): δ 3·85–3·95 (9H, 3 × *s*, 3 × OMe), 5·20 (1H, *dd*, J_{AB} 2Hz, J_{AC} 10Hz, CH_A), 5·65 (1H, *dd*, J_{BA} 2Hz, J_{BC} 18Hz, CH_B), 6·58 (1H, *s*, H-Ar), 7·05 (1H, *dd*, J_{CA} 10Hz, J_{CB} 18Hz, CH_C), 7·10 (1H, *s*, H-Ar).

The compound was identified as 2,4,5-trimethoxystyrene (**1a**) on the basis of the following evidence. The ABC signal in the PMR spectra was in close agreement with previously published data for the vinyl side-chain of styrenes [2] thus indicating the compound was a trimethoxy-substituted styrene. The absence of *ortho* coupling between the aromatic signals restricted the substitution pattern to three possibilities (**1a**–**1c**). Further PMR studies showed that, on the addition of the lanthanide shift reagent $Eu(fod)_3$, marked downfield shifts occurred in two of the OMe and both the H-Ar signals. We have observed [3], in studies on the effect of $Eu(fod)_3$ on coumarins, that complexation will occur between the reagent and two adjacent OMe groups but will not occur with single OMe substituents. The requirement for adjacent OMe substituents limits the choice therefore to **1a** or **1b**. The additional observation that there was an equal effect on the two H-Ar protons favours **1a** in which the complexing centre is adjacent to both rather than **1b** where only one is in close proximity.



(**1a**) –2,4,5-trimethoxy
(**1b**) –2,3,5-trimethoxy
(**1c**) –2,4,6-trimethoxy

Unequivocal proof of the structure was obtained by its synthesis from 2,4,5-trimethoxyacetophenone by the initial reduction of the acetophenone to the corresponding carbinol with $NaBH_4$ in boiling EtOH followed by dehydration of the carbinol at 240° with alumina [4]. Soxhlet extraction of the alumina with Et_2O followed by recrystallisation from aq. Me_2CO gave 2,4,5-trimethoxystyrene identical in all respects (UV, IR, MS, TLC, mmp) with the material isolated from *Pachypodanthium staudtii.*

Similar extraction of other plant parts revealed the occurrence of correspondingly large amounts of the styrene in the stem bark but much smaller quantities in the wood and leaf.

Biological significance. 2,4,5-Trimethoxystyrene has previously only been recorded as a tentatively identified constituent of the leaves of *Peperomia pellucida* HBK (Piperaceae) [5].

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* Part 1 in the projected series *Chemical Studies on the Annonaceae.*