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OCIMIN, A NOVEL NEOLIGNAN FROM *OCIMUM AMERICANUM*

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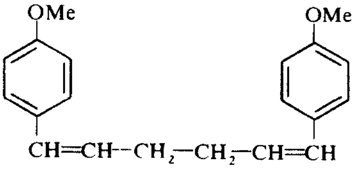
Key Word Index—*Ocimum americanum*; Labiatae; essential oil; methyl chavicol; 1,6-bis (4′-methoxy phenyl)-(E), (E)-hexa-1,5-diene; novel neolignan.

During the course of development of *Ocimum* species for useful essential oil constituents, a new strain of *O. americanum* L. (methyl chavicol-type) was developed by selection and hybridization. The essential oil of this new strain, which contained *ca* 70% methyl chavicol, was subjected to fractional distillation to remove all the methyl chavicol. On chilling, a solid was deposited which on repeated crystallization from EtOAc gave white fluorescent flakes, mp 158–161°. This compound was analysed for $C_{20}H_{22}O_2$, M^+ 294. The UV spectrum (MeOH) showed λ_{max} 265, 273 and 297 nm and the IR (nujol) showed characteristic bands for a *trans* double bond at 1610, 965 and 950 cm^{-1}. The 60 MHz ^1H NMR ($CDCl_3$) accounts for only 11 protons indicating that the molecule is a symmetrical dimer. It showed methoxyl protons at δ 3.8 and a broad triplet at 2.4 assigned to allylic methylene. Two AB doublets at δ 6.27 and 6.73 (J = 8 Hz) are due to 4 aromatic protons. One olefinic proton appeared as a doublet at 6.41 (J = 16 Hz) indicating the *trans* nature of the double bond. The other olefinic proton occurred as a complex multiplet between 5.97 and 6.33.

On hydrogenation, the new compound yielded a product having mp 68° (lit. 70–71°, reported as a by-product during synthesis of podocarpatriene) [4] analysed for $C_{20}H_{26}O_2$, M^+ 298. ^1H NMR showed signals at δ 1.4 (4H, *s*, CH_2—CH_2), 2.5 (2H, *t*, benzylic CH_2 –), 3.8 (3H, *s*, OMe) and 6.7–7.33 (H, *dd*, aromatic). Thus structure **1** was assigned on the basis of above data which was further confirmed by oxidation. On oxidation with $KMnO_4$, **1** gives two acids, identified as succinic acid and *p*-methoxy benzoic acid. The acids were identified by PC and ^1H NMR.



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EXPERIMENTAL

Material. Seeds of *O. americanum* were collected from Haldwani (Nainital, India) and raised in the nursery of R.R.L., Jammu [1–3]. A specimen is deposited with Kew Gardens (voucher 4901) and R.R.L., herbarium voucher 54324.

Isolation. Essential oil was obtained by steam distillation of the fresh green plant in a yield of 3.5% wt. The essential oil was fractionated on a Tower column 0.9 m long, 25 theoretical plates, and a reflux ratio of 10:1. After the isolation of methyl chavicol and linalool the residual fraction was chilled. The solidified material was filtered and crystallized from EtOAc to give a crystalline solid, mp 168–171°, analysed for $C_{20}H_{22}O_2$ (C, 81.32; H, 7.45).

Hydrogenation. The compound (50 mg) in EtOAc was hydrogenated over 5% Pd/C to give a solid, mp 68°.

Oxidation. $KMnO_4$ (1 g) was added to the compound (0.2 g) dissolved in Me_2CO (50 ml). The contents were refluxed for 3 hr on a H_2O bath. MnO_2 was filtered, the filtrate acidified and finally concd *in vacuo* to give a crude solid. Succinic acid (R_f 0.63) and *p*-methoxy benzoic acid (R_f 0.75) were identified by PC (*n*-BuOH–5% HOAc, 1:1) by comparison with reference samples. Fractional crystallization of the crude solid from Et_2O gave two acids. These were identified as *p*-methoxy benzoic acid, mp 179–181° (^1H NMR in TFA 3.71 for methoxyl proton and 2 doublets at 6.60 and 7.63, aromatic proton) and succinic acid, mp 182°.

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REFERENCES

1. Pushpangadan, P., Sobti, S. N. and Khan, R. (1975) *Nucleus* **18**, 177.
2. Sobti, S. N., Pushpangadan, P. and Atal, C. K. (1976) *Indian Perfum.* **12**, 59.
3. Sobti, S. N., Pushpangadan, P., Thappa, R. K., Aggarwal, S. G., Vashist, V. N. and Atal, C. K. (1978) *Lloydia* **41**, 50.
4. Delobelle, J. and Fetizon, M. (1961) *Bull. Soc. Chim. Fr.* 1894.