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SHORT REPORTS

STRUCTURE OF NEW AMIDE FROM *PIPER OFFICINARUM*

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Key Word Index—*Piper officinarum*; Piperaceae; *N*-isobutyl-trideca 13-(3,4, methylenedioxyphenyl)-2,4,12-trienamide.

We have reported the presence of methyl piperate in the fruits of *Piper officinarum* [1] and now wish to report the structure of a new amide from the same source. The petrol extract of the fruits, on repeated column chromatography over neutral alumina, gave crystalline needles mp 112–113. The compound analysed for $C_{24}H_{33}NO_3$ (found C, 75·20; H, 8·75; N, 3·79; calc. for $C_{24}H_{33}NO_3$: C, 75·19; H, 8·62 and N, 3·66%). UV(MeOH) λ_{max} 259 nm, indicating the probable presence of a conjugated system related to sorbamide [2,3]. IR (KBr) showed characteristic bands for –NH, (3290, 3060 cm^{-1}), –C=O (1625 cm^{-1}), –C=C– (1660, 1620 cm^{-1}), $-[CH=CH]_2-$, (997 cm^{-1}) and no peak in the region 960–965 cm^{-1}; indicating the presence of *trans* 2-*trans*-4 dienamide system [2,3]. The NMR ($CDCl_3$) spectrum showed a large doublet 0·9 δ (6H, $-C(C\underline{H}_3)_2$, *J*, 6Hz), a broad singlet at 1·37 (8H, $[CH_2]_4$), a multiplet between 1·9–2·4 (4H, allylic methylenes), a triplet at 3·19 (2H, $N-CH_2-C-$) typical for isobutyl amides; a singlet at 5·9 (2H, $O-H_2C-O$), multiplets between 5·7 to 6·2 (5H, olefinic protons, conjugated), doublet at 6·8 (3H, aromatic protons) and between δ 7–7·4 (IH, olefinic proton).

MS of the compound with fragments at 383, (M^+), *m/e* 161, 152, 73 and 148 supported its structure as the isobutylamide of 13-(3,4-methylenedioxy-phenyl)-tridec-2,4,12-enoic acid. On hydrogenation (Pd/c) the compound quickly absorbed 3 moles of H_2 to give the hexahydro derivative, mp 84–85° which on hydrolysis with HCl in alcohol in a sealed tube, gave isobutylamine hydrochloride mp 172°. The compound is therefore identified as *N*-isobutyl-trideca 13-(3,4, methylenedioxyphenyl): 2,4,12-trienamide.

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SESQUITERPENE ALCOHOLS IN CAMPHOR OIL

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Key Word Index—*Cinnamomum camphora*; Lauraceae; cadinenol; epicubenol; sesquiterpene alcohols.

Previous work. Non-volatile sesquiterpenoids in the leaves of the Camphor Tree [1].

Present work. Sesquiterpene alcohols have been investigated in camphor oil by many previous workers; the following were isolated from "blue oil" [2] which was prepared by the distillation of camphor oil at high temperature: α-cadinol, elemol, guaiol, β-eudesmol, juniper camphor, kusunol, campherenol and campherenone (ketone) [3–5]. Such conditions, however, may bring about thermal isomerization, dehydration, dehydrogenation, etc. to thermolabile components, so we have reinvestigated camphor oil which was isolated under less drastic conditions.

Monoterpenes, camphor and safrole were removed from the oil by distillation under reduced pressure (below 100°) and the residue was fractionated by chromatography on Si gel with *n*-hexane containing 0 ~ 50% EtOAc, preparative GLC, and TLC on Si gel; it was