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*N-ISO*BUTYLOCTADECA-*TRANS*-2-*TRANS*-4-DIENAMIDE: A NEW CONSTITUENT OF *PIPER GUINEENSE**

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Key Word Index—*Piper guineense*; Piperaceae; dienamide; *N-iso*butyloctadec-*trans*-2-*trans*-4-dienamide.

Piper guineense, West African Black Pepper or Ashanti Pepper, is a woody climber widely distributed throughout West Africa [1]. The fruits have been used as a flavorant while preparations of leaves, roots and seeds have been used internally as medicinal agents for the treatment of bronchitis, gastrointestinal disease and upset, venereal disease, and rheumatism [1]. Externally, preparations of the seeds have been used for their counterirritant and insecticidal properties [1].

P. guineense is reported to contain the lignans (+)-sesamin and (+)-ashantin [2, 3] but no other compounds have been isolated. *Iso*butylamides have to date been reported mainly in the Compositae [4] and Rutaceae [5]. However, *N-iso*butyldeca-*trans*-2-*trans*-4-dienamide has recently been isolated from the seeds of *Piper sylvaticum* Roxb. [6, 7]. This paper reports the isolation and identification of *N-iso*butyloctadeca-*trans*-2-*trans*-4-dienamide, a new naturally occurring *iso*butylamide from *P. guineense*.

Extraction of the seeds with acetone afforded an oily extract which was partitioned between Et_2O and HCl (1%). The Et_2O layer was shaken with $NaHCO_3$ (2%) and subsequently chromatographed over silicic acid. Elution with petrol–$CHCl_3$ (1:1) afforded *N-iso*butyloctadeca-*trans*-2-*trans*-4-dienamide as needles, m.p. 78–80°. The UV spectrum showed λ_{max}^{MeOH} 260 nm ($\log \epsilon$ 4·47) and was indicative of a sorbic (2,4-hexadienoic acid) chromophore [8]. The IR spectrum: (**1**) (M − Me_2CH, 8%); (**2**) (M − $NHCH_2CHMe_2$, 66%); (**3**) (M − $C_{12}H_{25}$, 22%); and (**4**) (M − $C_{13}H_{27}$, 30%) indicated the presence of a N–H group (3295 cm^{-1}), an $\alpha,\beta,\gamma,\delta$-conjugated carbonyl (1622 cm^{-1}) and a *trans*–*trans* conjugated diene system (1652 and 995 cm^{-1}) and was therefore suggestive of an $\alpha,\beta,\gamma,\delta$-unsaturated secondary amide (–C=C–C=CO–NH–). The MS showed M^+ at *m/e* 335 (100%) for $C_{22}H_{41}NO$ and other important fragments at 292 (M–Me_2CH, 8%) (**1**), 263 (**2**), 180 (**3**), and 166 (**4**). NMR: δ 0·93 (9H, m), $C\underline{H}_3$; 1·30 (22H, br, *s*) $C\underline{H}_2$; 1·65–2·20 (3H, m) $C\underline{H}$Æ and CH_2 next to conj. double bond; 3·25 (2H, *t*, J=6 Hz) $C\underline{H}_2$–N; 5·20–6·10 (4H, m) N$\underline{H}$ and $C\underline{H}$=; 6·90–7·40 (1H, m) $C\underline{H}$ in centre of conj. system. The

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* Part 6 in the series 'Constituents of West African Medicinal Plants'. For Part 5, see Tackie, A. N., Dwuma-Badu, D., Dabra, T. T., Knapp, J. E., Slatkin, D. J. and Schiff, P. L., Jr. (1974). Accepted for publication *Experientia*.

NMR ($CDCl_3$–60 MHz) and MS were in agreement with **1**. Hydrogenation of **1** over Adams catalyst in EtOH afforded a tetrahydroderivative (**2**), m.p. 74–75°; no absorption in the UV; ν_{max}^{KBr} 3310 cm^{-1} (NH) and 1640 cm^{-1} (CO); MS M^+ *m/e* 339 (15%) for $C_{22}H_{45}NO$, 296 (9), 282 (2), 267 (16) and 115 (100) which was identical (UV, IR, NMR, MS, m.p., m.m.p.) to an authentic sample of *N-iso*butyloctadecanamide (**2**) prepared according to Jacobson [9].

EXPERIMENTAL

M.ps are uncorrected. The IR spectra were in KBr and UV spectra in MeOH. The NMR spectra were determined at 60 MHz spectrometer in $CDCl_3$ with TMS as internal standard.

Isolation. The detailed isolation procedure of (**1**) and other constituents of *P. guineense** will be reported in due course. *N-iso*butyloctadeca-*trans*-2-*trans*-4-dienamide, white needles (decomposing on prolonged exposure to air); m.p. 78–80° petrol–EtOAc); $[\alpha]_D$ 0° (*c* 0·1, EtOAc); ν_{max}^{KBr} 3295 cm^{-1}, 2912, 2842, 1652, 1622, 1610, 1540, 1465, 1390, 1370, 1335, 1320, 1258, 1200, 1185, 1160, 998, 940, 875, 820, 720 and 660; MS M^+ *m/e* 335 (100%), 320 (30), [$335 \xrightarrow{m^*} 320$, m^* obs 305·5 and m^* calc 305·67], 306 (3), 292 (8), 280 (9), 279 (22), 264 (41), 263 (66), 250 (5), 236 (5), 222 (5), 220 (4), 208 (4), 194 (5), 180 (22), 167 (10), 166 (30), 154 (15), 153 (18), 152 (50), 138 (8), 126 (36), 115 (31), 110 (31), 57 (52) and 43 (52).

Catalytic hydrogenation in EtOH over PtO_2 for 6 hr under H_2 gave *N-iso*butyloctadecanamide (**2**) as white needles, m.p. 74–75°; λ_{max}^{MeOH} no absorption; ν_{max}^{KBr} 3310 cm^{-1}, 2919, 2843, 1640, 1545, 1470, 1383, 1260, 1240, 1222, 1205, 1190, 1160, and 720; $\delta_{60MHz}^{CDCl_3}$ δ0·93 (9H, *m*), 1·22 (30H, *br*, *s*), 1·72–2·27 (3H, *m*), 3·09 (2H; *t*, *J*–6·0 Hz), 5·50–5·55 (1H, *br*, *s*); MS M^+ *m/e* 339 (15%), 324 (2), 310 (2), 296 (9), 285 (5), 284 (26), 282 (1), 268 (5), 267 (16), 254 (1), 240 (1), 226 (1), 212 (1), 198 (2), 184 (6), 170 (7), 156 (2), 142 (8), 129 (9), 128 (63), 116 (22), 115 (100), 57 (64) and 43 (60).

*Preparation of N-*iso*butyloctadecanamide* [9]. Isobutyl amine (1·5 g) in Et_2O (10 ml) was treated with stearyl chloride (octadecanoyl chloride) from stearic acid (140 mg) with $SOCl_2$ (90 mg) in Et_2O (10 ml) slowly with stirring. After 1 hr the precipitated amine HCl-ide was worked up in the usual way to give white needles of *N-iso*butyloctadecanamide (84 mg), m.p. 74–76°, identical with (**2**) by direct comparison (UV, IR, NMR, MS, m.p., m.m.p.).

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* The plant material used in this study was collected in Ghana. Voucher specimens are on deposit at the Faculty of Pharmacy, University of Science and Technology, Kumasi, Ghana, West Africa.

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5-ALKOXY-FURANOCOUMARINS FROM *PEUCEDANUM OSTRUTHIUM**

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Plant. Peucedanum ostruthium (L.) Koch (syn. *Imperatoria ostruthium* L.) roots collected in South Tyrol (Italy). *Uses.* Medicinal—Radix Imperatoriae. *Previous work.* From the roots, several coumarins, furanocoumarins, a chromone derivative (peucenin) and hesperidin have been isolated [1, 2].

Present work. Dried roots (440 g) were extracted with C_6H_6 followed by MeOH. The residue from the methanolic extract afforded by separation on