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DEHYDRO-IPOMEAMARONE FROM INFECTED *IPOMOEA BATATAS* ROOT TISSUE

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When infected with *Ceratocystis fimbriata*, sweet potato root tissue accumulates in the infected region various kinds of furanoterpenoids such as ipomeamarone, ipomeamaronol and ipomeanine.[1–3] These terpenoids have an inhibitory action against the invading fungus.[4,5] Recently the presence of a new furano-terpenoid, dehydro-ngaione has been reported from *Athanasia crithmifolium* L.[6] In this communication, the authors report the isolation of a new compound from *Ipomoea batatas* having the same structure as dehydro-ngaione but different optical rotation.

The compound was isolated as a light yellow oil by repeated fractional column chromatography over silica gel from the $CHCl_3$–MeOH extracts of the dried, powdered sweet potato root tissue infected with *C. fimbriata*, and further purified by TLC and GLC. The compound was identical (IR, UV, NMR, MS, ORD, GLC and TLC, color reagent: 10% *p*-dimethylaminobenzaldehyde in 95% EtOH–conc.HCl, 1:1) with an authentic sample of dehydro-ngaione. However, it had a different ORD spectrum (dehydro-ipomeamarone, $[\alpha]_D^{21}$ + 14·7 (*c* 0·12, EtOH); dehydro-ngaione, $[\alpha]_D^{21}$ − 14·1 (*c* 0·42, EtOH) and from the relation between ngaione and ipomeamarone,[7,8] we have called the new compound dehydro-ipomeamarone.

EXPERIMENTAL

Roots of sweet potato (*Ipomoea batatas* L., variety, Norin No. 1) were harvested at the Kariya Farm, Aichi Prefecture in the autumn and stored at about 10° until used. The roots were cut into slices (2–3 mm thick) and both the cut surfaces inoculated with a spore suspension of *C. fimbriata*. After incubation for 1 week at 30°, the tissues were dried at room temperature and powdered. The powder (2·7 kg) was extracted with $CHCl_3$–MeOH (1:1). The extracts (crude oily substance, 180 g) were subjected to column chromatography over silica gel, using *n*-hexane–EtOAc giving dehydro-ipomeamarone (2·5 g). The compound was purified by TLC (Kiesel-gel HF 254,

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with *n*-hexane–EtOAc, 4:1 or 9:1) as described previously,[9] followed by GLC, using a column (2 m × 3 mm) of 10% 1,4-butanediol-succinate on shimalite (60–80 mesh) at 185°, with He gas (60 ml/min).

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ALKANES AND TRITERPENOIDS OF *QUERCUS GLAUCA*

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Plant. Quercus glauca Thunb. (Fagaceae) (Japanese name, arakashi). *Source.* Hiroshima Prefecture, Japan. *Uses.* Timber. *Previous work.* D-mannitol from the wood.[1]

Present work. The *n*-hexane soluble constituents of trunk-wood and leaves of the plant have been examined.

Trunk-wood. The chipped trunk-wood (22·8 kg) was extracted with MeOH (100 l.) at room temp. for 4 months. A part (5 l.) of the extract was concentrated to dryness to give a dark viscous oil (30·2 g), which was suspended over H_2O and extracted with *n*-hexane. The *n*-hexane soluble fraction (4·2 g) was chromatographed on a silica gel column with *n*-hexane–EtOAc as eluent with increasing EtOAc concentration, giving in order normal alkanes (417 mg), sitosterol (86 mg), and stigmast-4-en-3-one (82 mg).

n-*Alkanes.* GLC indicated the alkanes to be composed of alkanes having less carbon atoms than those of the leaves. *Sitosterol.* $C_{29}H_{50}O$; m.p. 138–139°; $[\alpha]_D^{25}$ −32·1° (*c* 0·78, $CHCl_3$); ν_{max} (Nujol) 3400 (OH), 1640 (C=C), 1050 cm^{-1} (C–O); δ_{ppm} ($CDCl_3$) 5·35 (*m*, 1H, $>C=C<^{H}$), 3·5 (*m*, 1 H, $>C\underline{H}(OH)$); direct comparison (TLC, IR, NMR, and m.m.p.) with an authentic sample. *Stigmast-4-en-3-one.* $C_{29}H_{48}O$; m.p. 89–90°; $[\alpha]_D^{25}$ +86·2° (*c* 4·12, $CHCl_3$); *m/e* 412·3753 (M^+, calcd. 412·3705); ν_{max} (KBr) 1660 (conjugated C=O), 1620 cm^{-1} (C=C); λ_{max} (EtOH) 245 nm (log ϵ 4·3), 240 (sh.), (Isooctane) 238 (3·95), 244 (sh.); δ_{ppm} ($CDCl_3$ 5·72 (s, 1 H, $>C=C<^{H}$), 2·30 (m, 2H, $-CO-C\underline{H}_2-$). These data suggested the conjugated carbonyl compound to be stigmast-4-en-3-one; this was confirmed with an authentic specimen (co-TLC, IR, NMR, MS, and m.m.p.) prepared[2] from sitosterol by the Oppenauer oxidation.

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