

SHORT COMMUNICATION

ISOLATION OF HOPENONE-B FROM *EUPHORBIA CYPARISSIAS**

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Abstract—The occurrence in nature of hopenone-b, isolated from *Euphorbia cyparissias*, is reported for the first time.

SEVERAL triterpenoids have been reported¹ as constituents of *Euphorbia cyparissias* L. (Euphorbiaceae). A further investigation of the fraction yielding glut-5(6)-en-3-one has resulted in the isolation of another ketone which exhibited physical and spectral properties characteristic of hopenone-b.^{2,3} Dehydration² of hydroxyhopenone from Dammar gum⁴ gave a mixture of two isomers from which hopenone-b was isolated and demonstrated to be identical with the ketone from *E. cyparissias*. LiAlH_4 reduction of hopenone-b gave hop-22(29)-en-3 β -ol as the major product. This is the first report of the occurrence in nature of hopenone-b. Moretenone, differing only in the orientation of the side-chain, has been reported from *E. lateriflora*.⁵

EXPERIMENTAL

M.ps. are uncorrected and were determined in sealed capillary tubes using a Büchi melting point apparatus. PMR spectra were obtained in CDCl_3 with a Varian A-60A spectrometer using TMS as internal standard. The mass spectrum was obtained with a Varian M-66 mass spectrometer (ionizing energy 70 eV).

Isolation and Identification of Ketone

Chromatography of material (220 mg) from mother liquors¹ of glut-5(6)-en-3-one on AgNO_3 -Kieselgel plates gave additional glut-5(6)-en-3-one and a second ketone (68 mg) which on recrystallization (CHCl_3 -acetonitrile) yielded plates, m.p. 220–221°; $[\alpha]_D^{25} + 85^\circ$ (c, 0.80 in CHCl_3); positive Zimmerman test; ν_{max} (KBr) 1701 cm^{-1} ($-\text{C}=\text{O}$), 1638 and 883 cm^{-1} ($-\text{C}=\text{CH}_2$); PMR spectrum: δ 0.75 (3H), 0.96 (3H), 1.03 (6H), 1.08 (3H), 1.77 (3H), 4.81 (multiplet; 2H); mass spectrum: m/e 424 (M^+), 409 ($\text{M}-\text{CH}_3$), 381 (M -side chain), 205 (base peak), 189. (Found: C, 84.86; H, 11.24. Calc. for $\text{C}_{30}\text{H}_{48}\text{O}$: C, 84.84; H, 11.39 per cent.) This compound could be distinguished chromatographically (AgNO_3 -Kieselgel plates) from moretenone and zeorininone-b kindly supplied by Dr. H. N. Khastgir and Dr. I. Yosioka respectively.

* Contribution No. 412.

¹ A. N. STARRATT, *Phytochem.* **5**, 1341 (1966).

² W. J. DUNSTAN, H. FAZAKERLEY, T. G. HALSALL and E. R. H. JONES, *Croat. Chem. Acta* **29**, 173 (1957).

³ Y. TSUDA and M. HATTORI, *Chem. Pharm. Bull.* **15**, 1073 (1967).

⁴ J. S. MILLS and A. E. A. WERNER, *J. Chem. Soc.* 3132 (1955).

⁵ D. LAVIE, M. K. JAIN and T. O. OREBAMJO, *Phytochem.* **7**, 657 (1968).

Hydroxyhopanone obtained⁴ from Dammar gum was dehydrated using POCl_3 in pyridine.² The product, a mixture of hopenone-a and hopenone-b, was separated by chromatography on AgNO_3 -Kieselgel plates. Hopenone-b from the slower-moving band crystallized from CHCl_3 -acetonitrile as plates, m.p. 219–222°, $[\alpha]_D + 86^\circ$ (c, 0.65 in CHCl_3), and was identical (mixture m.p., i.r. PMR, TLC comparisons) with the ketone from *Euphorbia cyparissias*. Tsuda and Hattori³ report m.p. 219–220° for this compound.

Hydride Reduction of Hopenone-b

Hopenone-b (12 mg) in anhydrous ether (10 ml) was heated under reflux for 2 hr with LiAlH_4 (15 mg). The major product was isolated by separation on Kieselgel plates and recrystallized from CHCl_3 -acetonitrile to give hop-22(29)-en-3 β -ol, m.p. 255–261°; ν_{max} (KBr) 3488, 1638, 886 cm^{-1} . (Found: C, 84.40; H, 11.96. Calc. for $\text{C}_{30}\text{H}_{50}\text{O}$: C, 84.44; H, 11.81 per cent.)

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