X-Ray Structure of Lyofoligenic Acid, a Novel B-Homotriterpene

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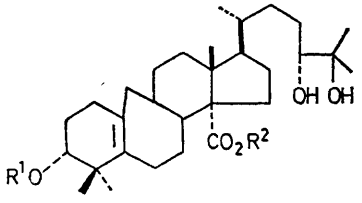
Summary The structure of lyofoligenic acid has been determined as 3α,24,25-trihydroxy-9(10→19)-*abeo*-lanost-5(10)-en-32-oic acid (II) by chemical and spectroscopic studies as well as by *X*-ray crystallographic analysis of the *p*-bromobenzoate methyl ester (III) of (II).

FROM the leaves of *Lyonia ovalifolia* var. *elliptica* was isolated a new triterpene glucoside, lyofolic acid (I), which contains a cyclopropane ring. Acidic hydrolysis of (I) gave glucose and lyofoligenic acid (II), $C_{30}H_{50}O_5 \cdot \frac{1}{2}H_2O$, m.p.



(II) $R^1 = R^2 = H$
(III) R = *p*-BrC_6H_4CO, R^2 = Me

210—212°, $[\alpha]_D$ +72° (MeOH).[1] ^1H and ^{13}C n.m.r. spectral data for (II) showed no evidence for the presence of cyclopropane rings or further methyl groups, but indicated the presence of a tetrasubstituted double bond. Its structure was elucidated by chemical and spectroscopic studies, and its stereochemistry was confirmed by *X*-ray crystallographic analysis of the *p*-bromobenzoate methyl ester (III).

Single crystals of compound (III), $C_{41}H_{59}BrO_6$, m.p. 161·5—163·5° (EtOH), are orthorhombic, space-group $P2_12_12_1$, $Z = 4$, $a = 13{\cdot}576$, $b = 35{\cdot}386$, $c = 8{\cdot}068$ Å. 1576 independent structure factors were collected with a Philips automatic four-circle diffractometer using monochromated Mo-K_α radiation. The structure was solved by the heavy-atom method to a current *R* index of 0·09. The molecular structure and stereochemistry are shown in the Figure.

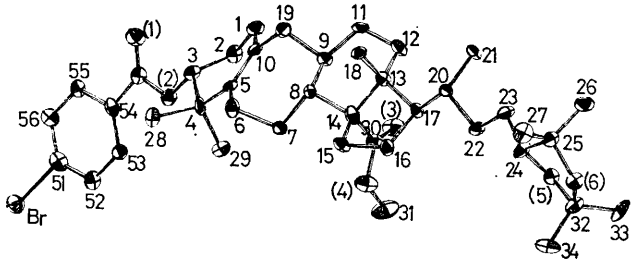


FIGURE. ORTEP[2] drawing of the molecular structure of compound (III) viewed along the *c* axis.

Compound (I) is the first recognized lanostane derivative carrying 3α-hydroxy and 14α-carboxy-groups, which suggest that removal of the 32-carbon atom in steroid biosynthesis[3] may occur from a carboxylic acid resulting in the release of carbon dioxide.

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