X-Ray Crystal Structure Analysis of Papyriogenin A

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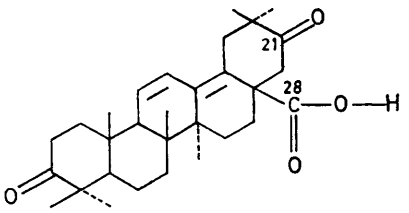
Summary The crystal structure of a new oleanane type triterpene, papyriogenin A, has been shown by *X*-ray analysis to be 3,21-dioxo-oleana-11,13(18)-diene-28-oic acid.

A NEW glycoside, papyrioside L-II, has been isolated from the leaves of *Tetrapanax papyferum* (Araliaceae). Acid hydrolysis of papyrioside L-II gave a new pentacyclic triterpene, papyriogenin A, having a heteroannular diene (u.v. 243, 252 and 260 nm), a carboxy and two carbonyl groups. The parent glycoside however shows only end absorption in its u.v. spectrum. As a preliminary to the determination of the structure of papyrioside L-II, the structure of the derived aglycone was determined by *X*-ray crystallography.



FIGURE

Crystal data: Papyriogenin A, m.p. 262—264 °C, $C_{30}H_{42}O_4$, space group $P2_12_12_1$, orthorhombic, $a = 18{\cdot}308$, $b = 21{\cdot}443$, $c = 6{\cdot}562$ Å and $Z = 4$. Intensity data were collected using Cu–K_α radiation and an automatic four-circle diffractometer and 1688 independent reflections were observed. The structure was solved by direct methods using the program MULTAN. Block-matrix, least-squares refinement anisotropic temperature factors for C and O and isotropic temperature factors for H converged to $R = 0{\cdot}053$ for observed reflections.

The structure (Figure) is fully consistent with the spectral and chemical data. The existence of the intermolecular hydrogen bond between OH(28) and O(21) in the crystalline state [distance between O(28) and O(21) 2·79 Å] inhibits the association of the carboxy group and explains the high frequency CO absorption at 1730 cm^{-1} compared with that of oleanolic acid (1690 cm^{-1}).

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