

THE STRUCTURE OF PENTAGYDINE, A NEW DITERPENOID ALKALOID

A.G. González, G. de la Fuente and R. Díaz

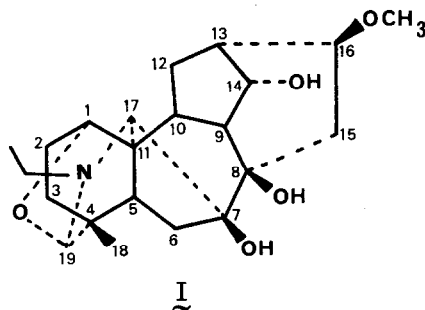
Instituto de Productos Naturales Orgánicos, C.S.I.C., La Laguna, Tenerife, Spain
and

P.G. Jones and G.M. Sheldrick

Institut für Anorganische Chemie, Tammannstrasse 4, D-3400 Göttingen, West Germany

Summary: Spectroscopic and X-ray data are given for pentagydine, a new diterpenoid alkaloid isolated from Delphinium pentagynum.

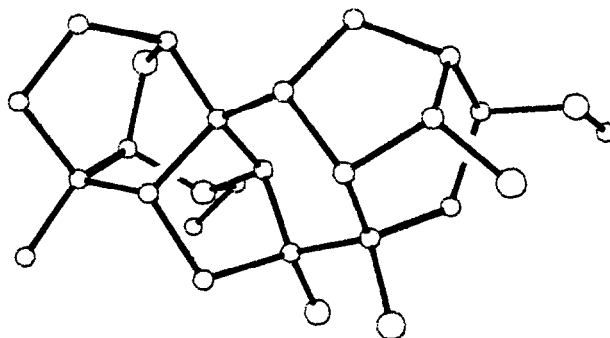
Continuing our study of diterpenoid alkaloids from the plant *Delphinium pentagynum* Lam¹ we have isolated a new base, pentagydine (I), as a minor constituent.



Pentagydine has m.p. 130-131°C and analysed for $C_{22}H_{33}NO_5$ by HRMS. Its PMR displayed signals at δ 0.88 (3H, s, CH_3), 1.11 (3H, t, $J = 7$ Hz, $N-CH_2-CH_3$), 3.38 (3H, s, OCH_3) and 4.20 (1H, t, $J = 4.5$ Hz, C-14H). The existence of the C-1-C-19 inner ether in the molecule was inferred from the IR absorptions at 990 and 885 cm^{-1} , the ion at $M^+-C_3H_4O$ (base peak) in the HRMS³ and the signals at δ 3.67 (1H, m, $W_{1/2} = 9$ Hz, C-18H) and 3.85 (1H, s, C-19H) in its PMR spectrum.

Biogenetic considerations and the oxygenation pattern of lycoctonine-type alkaloids⁴ permitted the tentative assignment of structure (I) to pentagydine, later confirmed by an X-ray diffraction experiment. The compound, $C_{22}H_{33}NO_5 \cdot H_2O$, crystallized in the monoclinic space group $P2_1$ with four molecules in a cell, $a = 8.542(2)$, $b = 23.564(6)$, $c = 10.117(3)$ Å, and $U = 2036.2$ Å³. The structure was solved by direct methods and refined to $R = 0.042$ and $R_w = 0.042$ for 4711

unique observed reflections (MoK α radiation, $2\theta_{\max} = 55^\circ$, Friedel opposites merged). No significant discrepancies were noted between the two crystallographically-independent molecules.



We have also isolated karakoline⁴, identified by comparison with an authentic sample. Pentagydine and karakoline may be considered respectively biogenetic precursors of gadesine and dihydropentagynine, also found in this plant¹.

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R E F E R E N C E S

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