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Structure of Delvestine: a Norditerpenoid Alkaloid from *Delphinium vestitum* Wall

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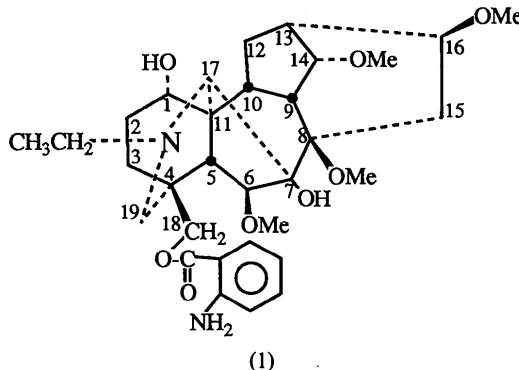
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Abstract. C₃₂H₄₆N₂O₈, *M_r* = 586.73, m.p. 458–460 K, monoclinic, *P*2₁, *a* = 9.187 (2), *b* = 14.979 (3), *c* = 11.474 (2) Å, β = 104.09 (2)°, *V* = 1531.5 (9) Å³, *Z* = 2, *D_x* = 1.27 g cm⁻³, λ (Cu *K* α) = 1.5418 Å, μ = 7.1 cm⁻¹, *F*(000) = 632, room temperature, *R* = 0.039, *wR* = 0.053 for 3077 observed reflections [*I* > 3 σ (*I*)]. The aminoethyl C(21) atom is disordered. There is an intramolecular hydrogen bond between O(1)—H(O1) and N(1) atoms, and between O(3)—H(O3) and O(4), stabilizing the boat conformations adopted by the rings *A* and *D*.

Introduction. The polyoxygenated norditerpenoid alkaloids of *Aconitum* and *Delphinium* species have been recognized since antiquity as poisonous towards animals and as possessing medicinal properties (Benn & Jacyno, 1983). In an earlier investigation, we have reported the isolation and structure determination of

delvestine (1) from *Delphinium vestitum* Wall (Desai, Joshi & Pelletier, 1985). We report here the X-ray crystallographic studies of delvestine to confirm the structure and stereochemistry of this norditerpenoid alkaloid as 4-[(2-aminobenzoyl)oxy]methyl]-20-ethyl-6 β ,8,16 β -tetramethoxyaconitane-1 α ,7-diol.



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Experimental. *Delphinium vestitum* was extracted with 85% ethanol. From this extract, the alkaloid was isolated by a combination of ion-exchange and vacuum liquid chromatography followed by centrifugally accelerated thin-layer chromatography (Chromatotron). Colorless crystals were obtained from hexane. Three-dimensional intensity data were collected on a CAD-4 diffractometer using a crystal of dimensions $0.3 \times 0.3 \times 0.5$ mm; the lattice parameters were refined using 25 centered reflections in the range $20 < \theta < 30^\circ$; reflections were measured ($2\theta \leq 150^\circ$) using $\omega/2\theta$ scan; ω scan width was calculated using the expression $(0.70 + 0.14 \tan \theta)^\circ$; the horizontal aperture width ranged from 2.4 to 3.5 mm and the vertical aperture width was set at 4.0 mm; range of hkl : h 0 to 11, k -18 to 18 and l -14 to 14; a total of 6721 reflections were measured; 3283 were unique of which 3077 reflections were significant [$I > 3\sigma(I)$]; $R_{\text{symm}} = 0.025$; three reflections were measured every hour of X-ray exposure and they showed 5.3% variation in their intensities during the course of data collection; a linear decay correction was applied; the correction factors on I ranged from 1.000 to 1.028 with an average value of 1.013; Lorentz and polarization and anisotropy of absorption using ψ -scan corrections (North, Phillips & Mathews, 1968) were applied to the data; the average, minimum and maximum transmissions are 0.915, 0.78 and 1.00, respectively.

The structure was solved by the application of direct methods using the program *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); the structural parameters for the non-H atoms were refined using full-matrix least-squares methods; the methyl C atom, C(21), of the aminoethyl group is disordered; the occupancy factors for the two disordered sites were initially set to 0.5 each; later refinement of the occupancies resulted in 0.65 for C(21A) and 0.35 for C(21B); all H atoms [except those attached to the disordered C(21A), C(21B) and one atom attached to C(25)] were located from a difference electron density map computed at an R of 8.5%; these H atoms except H1(C25) were then included in the refinement procedure with individual isotropic thermal parameters; the R value at the last cycle of refinement, with the non-H atoms treated anisotropically and the H atoms isotropically, was 0.039 for 3077 observed reflections; wR was 0.053; the function minimized in the least-squares procedure was $\sum w(|F_o|^2 - 1/k|F_c|^2)$ where $w = 4|F_o|^2/\sigma^2(|F_o|^2)$, $\sigma(|F_o|^2) = [\sigma(I) + s^2I^2]Lp$, $s = 0.02$, $\sigma(I)$ is based on intensity statistics and k is the scale factor; $(\Delta/\sigma)_{\text{max}} < 0.1$ for all atoms in the last cycle; largest feature in the difference electron density map at $R = 0.039$ was 0.20 and -0.15 e \AA^{-3} ; $S = 1.8$; atomic scattering factors from *International Tables for X-ray Crystallography*

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters of non-H atoms with e.s.d.'s in parentheses*

The equivalent isotropic displacement parameter, U_{eq} , is defined as $U_{\text{eq}} = \frac{1}{3}[U_{22} + 1/\sin^2\beta(U_{11} + U_{33} + 2U_{13}\cos\beta)]$.

	x	y	z	U_{eq} (\AA^2)
C(1)	0.6915 (2)	0.4015 (1)	0.0435 (2)	0.048 (3)
O(1)	0.5337 (1)	0.4204 (1)	0.0036 (1)	0.059 (3)
C(2)	0.7611 (2)	0.4491 (2)	0.1607 (2)	0.058 (4)
C(3)	0.7026 (2)	0.4106 (2)	0.2629 (2)	0.058 (4)
C(4)	0.7186 (2)	0.3068 (2)	0.2714 (1)	0.050 (4)
C(5)	0.8072 (2)	0.2696 (1)	0.1817 (1)	0.042 (3)
C(6)	0.7986 (2)	0.1668 (1)	0.1782 (1)	0.044 (3)
O(2)	0.9389 (1)	0.1262 (1)	0.1777 (1)	0.058 (3)
C(22)	1.0336 (3)	0.1175 (3)	0.2931 (2)	0.090 (7)
C(7)	0.6677 (2)	0.1448 (1)	0.0651 (1)	0.039 (3)
O(3)	0.5688 (1)	0.0817 (1)	0.0969 (1)	0.058 (3)
C(8)	0.7261 (2)	0.1114 (1)	-0.0436 (1)	0.042 (3)
O(4)	0.7776 (2)	0.0222 (1)	-0.0033 (1)	0.055 (3)
C(23)	0.9021 (3)	-0.0162 (2)	-0.0313 (3)	0.124 (7)
C(9)	0.8467 (1)	0.1765 (1)	-0.0654 (1)	0.039 (3)
C(10)	0.8101 (1)	0.2760 (1)	-0.0445 (1)	0.039 (3)
C(11)	0.7245 (1)	0.299	0.0529 (1)	0.037 (3)
C(12)	0.7217 (2)	0.3100 (2)	-0.1714 (1)	0.049 (4)
C(13)	0.7081 (2)	0.2296 (2)	-0.2552 (1)	0.051 (4)
C(14)	0.8548 (2)	0.1811 (2)	-0.1978 (1)	0.047 (3)
O(5)	0.8795 (2)	0.0965 (1)	-0.2455 (1)	0.060 (3)
C(24)	0.9143 (3)	0.1022 (3)	-0.3581 (2)	0.086 (6)
C(15)	0.6007 (2)	0.0975 (2)	-0.1590 (2)	0.051 (4)
C(16)	0.5698 (2)	0.1721 (2)	-0.2532 (2)	0.054 (4)
O(6)	0.5137 (2)	0.1267 (2)	-0.3655 (1)	0.081 (5)
C(25)	0.4132 (5)	0.1743 (4)	-0.4485 (3)	0.15 (1)
C(17)	0.5892 (1)	0.2365 (1)	0.0357 (1)	0.039 (3)
N(1)	0.4917 (1)	0.2662 (1)	0.1141 (1)	0.047 (3)
C(20)	0.3407 (2)	0.2254 (2)	0.0884 (2)	0.067 (5)
C(21A)	0.2582 (3)	0.2268 (5)	-0.0434 (4)	0.10 (2)
C(21B)	0.2390 (6)	0.274 (1)	0.1450 (7)	0.10 (3)
C(19)	0.5640 (2)	0.2615 (2)	0.2424 (1)	0.053 (4)
C(18)	0.7955 (2)	0.2826 (2)	0.4009 (2)	0.066 (6)
O(7)	0.9464 (2)	0.3171 (2)	0.4262 (1)	0.074 (5)
C(26)	1.0083 (3)	0.3509 (2)	0.5346 (1)	0.062 (5)
O(8)	0.9400 (2)	0.3524 (2)	0.6119 (1)	0.104 (5)
C(27)	1.1599 (3)	0.3841 (2)	0.5445 (2)	0.059 (4)
C(28)	1.2462 (3)	0.4183 (2)	0.6554 (2)	0.069 (5)
N(2)	1.1931 (3)	0.4209 (2)	0.7563 (2)	0.095 (7)
C(29)	1.3890 (3)	0.4504 (3)	0.6593 (2)	0.082 (7)
C(30)	1.4471 (3)	0.4509 (3)	0.5597 (3)	0.093 (9)
C(31)	1.3620 (3)	0.4180 (3)	0.4502 (2)	0.088 (8)
C(32)	1.2210 (3)	0.3852 (2)	0.4442 (2)	0.070 (6)

(1974); program for least-squares refinement, Fourier analysis and *ORTEPII* (Johnson, 1976) as in *SDP* (Frenz, 1982).

Discussion. The final positional and equivalent isotropic thermal parameters for all atoms are listed in Table 1.* The bond lengths and angles are given in Table 2. The conformation adopted by the molecule is shown in Fig. 1. The central ring system of delvestine is formed by the fusion of four six-membered rings and two five-membered rings. The individual rings comprising delvestine adopt a slightly different conformation than the rings of acoforestine (Pelletier, Joshi, Glinski, Chokshi, Chen, Bhandary

* Lists of structure factors, anisotropic thermal parameters, H-atom positions, bond lengths and angles involving H atoms, torsional angles and results of least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52859 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (Å) and bond angles (°) with *e.s.d.*'s in parentheses

C(1)—O(1)	1.439 (2)	C(12)—C(13)	1.528 (3)
C(1)—C(2)	1.519 (3)	C(13)—C(14)	1.531 (2)
C(1)—C(11)	1.562 (2)	C(13)—C(16)	1.539 (3)
C(2)—C(3)	1.517 (3)	C(14)—O(5)	1.421 (3)
C(3)—C(4)	1.563 (4)	O(5)—C(24)	1.407 (3)
C(4)—C(5)	1.562 (2)	C(15)—C(16)	1.532 (3)
C(4)—C(19)	1.536 (3)	C(16)—O(6)	1.437 (3)
C(4)—C(18)	1.526 (2)	O(6)—C(25)	1.356 (5)
C(5)—C(6)	1.542 (3)	C(17)—N(1)	1.484 (2)
C(5)—C(11)	1.552 (2)	N(1)—C(20)	1.478 (2)
C(6)—O(2)	1.426 (2)	N(1)—C(19)	1.462 (2)
C(6)—C(7)	1.574 (2)	C(20)—C(21A)	1.519 (5)
O(2)—C(22)	1.403 (3)	C(20)—C(21B)	1.45 (1)
C(7)—O(3)	1.419 (2)	C(18)—O(7)	1.441 (3)
C(7)—C(8)	1.554 (2)	O(7)—C(26)	1.335 (2)
C(7)—C(17)	1.550 (3)	C(26)—O(8)	1.206 (3)
C(8)—O(4)	1.455 (2)	C(26)—C(27)	1.457 (3)
C(8)—C(9)	1.541 (2)	C(27)—C(28)	1.421 (3)
C(8)—C(15)	1.543 (2)	C(27)—C(32)	1.398 (3)
O(4)—C(23)	1.386 (4)	C(28)—N(2)	1.363 (3)
C(9)—C(10)	1.560 (3)	C(28)—C(29)	1.388 (4)
C(9)—C(14)	1.541 (2)	C(29)—C(30)	1.374 (4)
C(10)—C(11)	1.554 (2)	C(30)—C(31)	1.398 (4)
C(10)—C(12)	1.570 (2)	C(31)—C(32)	1.371 (4)
C(11)—C(17)	1.531 (2)		
O(1)—C(1)—C(2)	111.3 (2)	C(7)—C(8)—C(9)	109.2 (1)
O(1)—C(1)—C(11)	112.2 (1)	C(7)—C(8)—C(15)	113.6 (1)
C(2)—C(1)—C(11)	111.5 (1)	O(4)—C(8)—C(9)	116.2 (1)
C(1)—C(2)—C(3)	110.8 (2)	O(4)—C(8)—C(15)	105.4 (2)
C(2)—C(3)—C(4)	112.4 (2)	C(9)—C(8)—C(15)	111.2 (1)
C(3)—C(4)—C(5)	111.7 (2)	C(8)—O(4)—C(23)	122.0 (2)
C(3)—C(4)—C(19)	111.0 (2)	C(8)—C(9)—C(10)	112.9 (1)
C(3)—C(4)—C(18)	108.1 (2)	C(8)—C(9)—C(14)	113.5 (1)
C(5)—C(4)—C(19)	107.6 (1)	C(10)—C(9)—C(14)	99.9 (1)
C(5)—C(4)—C(18)	111.4 (2)	C(9)—C(10)—C(11)	119.2 (1)
C(19)—C(4)—C(18)	106.9 (2)	C(9)—C(10)—C(12)	104.5 (1)
C(4)—C(5)—C(6)	110.0 (1)	C(11)—C(10)—C(12)	110.9 (1)
C(4)—C(5)—C(11)	108.2 (1)	C(1)—C(11)—C(5)	112.8 (1)
C(6)—C(5)—C(11)	104.4 (1)	C(1)—C(11)—C(10)	107.0 (1)
C(5)—C(6)—O(2)	112.7 (1)	C(1)—C(11)—C(17)	117.1 (1)
C(5)—C(6)—C(7)	104.7 (1)	C(5)—C(11)—C(10)	113.9 (1)
O(2)—C(6)—C(7)	114.6 (1)	C(5)—C(11)—C(17)	98.2 (1)
C(6)—O(2)—C(22)	112.9 (2)	C(10)—C(11)—C(17)	108.0 (1)
C(6)—C(7)—O(3)	109.6 (1)	C(10)—C(12)—C(13)	105.8 (2)
C(6)—C(7)—C(8)	112.6 (1)	C(12)—C(13)—C(14)	100.1 (1)
C(6)—C(7)—C(17)	102.4 (1)	C(12)—C(13)—C(16)	111.9 (2)
O(3)—C(7)—C(8)	111.1 (1)	C(14)—C(13)—C(16)	111.9 (2)
O(3)—C(7)—C(17)	110.6 (1)	C(9)—C(14)—C(13)	101.3 (1)
C(8)—C(7)—C(17)	110.1 (1)	C(9)—C(14)—O(5)	112.9 (2)
C(7)—C(8)—O(4)	100.9 (1)	C(13)—C(14)—O(5)	117.6 (1)
C(14)—O(5)—C(24)	113.0 (2)	C(4)—C(18)—O(7)	107.8 (2)
C(8)—C(15)—C(16)	118.6 (2)	C(18)—O(7)—C(26)	119.6 (2)
C(13)—C(16)—C(15)	114.0 (1)	O(7)—C(26)—O(8)	120.8 (2)
C(13)—C(16)—O(6)	111.7 (2)	O(7)—C(26)—C(27)	112.6 (2)
C(15)—C(16)—O(6)	104.6 (2)	O(8)—C(26)—C(27)	126.5 (2)
C(16)—O(6)—C(25)	114.5 (3)	C(26)—C(27)—C(28)	120.5 (2)
C(7)—C(17)—C(11)	101.2 (1)	C(26)—C(27)—C(32)	120.5 (2)
C(7)—C(17)—N(1)	116.8 (1)	C(28)—C(27)—C(32)	119.0 (2)
C(11)—C(17)—N(1)	109.2 (1)	C(27)—C(28)—N(2)	122.3 (2)
C(17)—N(1)—C(20)	115.5 (2)	C(27)—C(28)—C(29)	118.1 (2)
C(17)—N(1)—C(19)	113.8 (1)	N(2)—C(28)—C(29)	119.6 (2)
C(20)—N(1)—C(19)	110.8 (2)	C(28)—C(29)—C(30)	122.0 (2)
N(1)—C(20)—C(21A)	114.2 (2)	C(29)—C(30)—C(31)	120.0 (3)
N(1)—C(20)—C(21B)	112.3 (5)	C(30)—C(31)—C(32)	119.1 (3)
C(21A)—C(20)—C(21B)	102.6 (4)	C(27)—C(32)—C(31)	121.8 (2)
C(4)—C(19)—N(1)	111.5 (2)		

[−0.828 (2) Å] the plane through the atoms C(4), C(11), C(17) and C(19). Ring *B* of acforestine is also a chair but the C(4) and C(17) atoms deviate from the plane through the remaining atoms. Ring *C* [C(7), C(8), C(9), C(10), C(11) and C(17)] is a distorted chair with C(9) below [−0.454 (1) Å] and C(17) above [0.875 (1) Å] the plane through the remaining four atoms. In acforestine, the atoms C(7) and C(10) deviate from the plane. Ring *D* [C(8), C(9), C(14), C(13), C(16) and C(15)] is a half-boat with C(14) and C(15) forming the end atoms above the plane through C(8), C(9), C(13) and C(16) by 0.841 (2) and 0.338 (2) Å, respectively. The conformation of the *D* ring in acforestine is almost the same with slight differences in the deviation of atoms C(14) (0.86 Å) and C(15) (0.26 Å) from the plane through the other four atoms. The five-membered ring *E* [C(5), C(6), C(7), C(17) and C(11)] is puckered with $\tau_m = 53.4 (1)^\circ$ and $\varphi = 254.4 (1)^\circ$ (Altona, Geise & Romers, 1968), and ring *F* [C(9), C(10), C(12), C(13) and C(14)] is in an envelope conformation with $\tau_m = 53.0 (1)^\circ$ and $\varphi = 88.0 (2)^\circ$. The corresponding values for acforestine are $\tau_m = 48.6^\circ$ and $\varphi = 301^\circ$ for ring *E* and $\tau_m = 53.7^\circ$ and $\varphi = 114^\circ$ for ring *F*.

The molecular and crystal structure is stabilized by both intra- as well as intermolecular hydrogen bonds (Table 3), thus utilizing all the donor H atoms. The boat conformation adopted by ring *A* is stabilized by an intramolecular hydrogen bond, a feature common in norditerpenoid alkaloids having an α -hydroxyl group at the C(1) position (Pelletier, Djarmati, Lajsic & DeCamp, 1976). There are also two other intramolecular hydrogen bonds [N(2)—H1(N2)⋯O(8) and O(3)—H(O3)⋯O(4)] of which the latter is a bifurcated hydrogen bond. The molecular packing is stabilized by two intermolecular hydrogen bonds [between O(3)—H(O3)⋯O(1) and N(2)—H2(N2)⋯O(4)]. The geometries of these hydrogen bonds are listed in Table 3. The X-ray crystal structures of

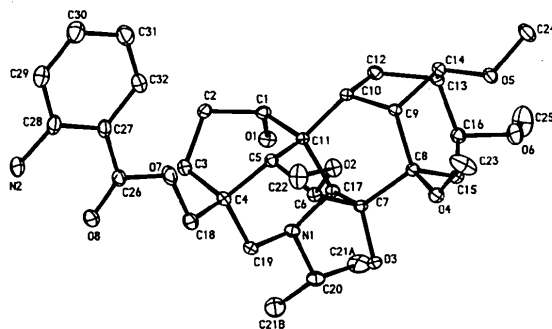


Fig. 1. A perspective view of the molecule showing the atomic numbering scheme. The thermal ellipsoids are at 25% probability level.

& Go, 1987) which also is made up of a similar fused ring system. Ring *A* [C(1), C(2), C(3), C(4), C(5) and C(11)] adopts a boat conformation with C(2) and C(5) atoms above the plane through the atoms C(1), C(3), C(4), C(11) by −0.655 (2) and −0.686 (1) Å, respectively. In acforestine, a norditerpenoid alkaloid containing an α -methoxy group at C(1), ring *A* is in a chair conformation. Ring *B* [C(4), C(5), C(11), C(17), N(1) and C(19)] is a distorted chair with N(1) above [0.628 (2) Å] and C(5) below

Table 3. *Geometry of the hydrogen bonds with e.s.d.'s in parentheses*

D	H	A	D—H (Å)	H...A (Å)	D...A (Å)	D—H...A (°)	Symmetry code
O(1)	H(O1)	N(1)	0.87 (3)	1.99 (4)	2.708 (3)	140 (3)	x, y, z
O(3)	H(O3)	O(4)	0.74 (3)	2.35 (2)	2.621 (2)	104 (2)	x, y, z
N(2)	H1(N2)	O(8)	0.92 (2)	2.03 (3)	2.708 (4)	129 (2)	x, y, z
O(3)	H(O3)	O(1)	0.74 (3)	2.03 (3)	2.744 (2)	161 (3)	$-x, -\frac{1}{2} + y, -z$
N(2)	H2(N2)	O(4)	0.99 (3)	2.30 (4)	3.167 (3)	145 (3)	$2 - x, \frac{1}{2} + y, 1 - z$

more than 30 norditerpenoid alkaloids have been determined and the absolute stereochemistry of some has been established (Joshi & Pelletier, 1987). The absolute configuration of delvestine is assumed by analogy with other alkaloids of this class.

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Structure of 2-*tert*-Butyl-3-phenyl-2-cyclopropenecarboxylic Acid

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Abstract. $C_{14}H_{16}O_2$, $M_r = 216.28$, triclinic, $P\bar{1}$, $a = 8.081$ (4), $b = 8.390$ (3), $c = 10.247$ (3) Å, $\alpha = 79.70$ (2), $\beta = 81.46$ (3), $\gamma = 72.56$ (3)°, $V = 648.8$ (5) Å³, $Z = 2$, $D_x = 1.11$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.709261$ Å, $\mu = 0.70$ cm⁻¹, $F(000) = 232$, $T = 293$ K, $R = 0.056$ for 2780 observed reflexions. The *tert*-butyl group of the title compound does not affect the coplanar arrangement of the phenyl ring and the cyclopropene double bond. The carboxyl group possesses a bisected conformation relative to the cyclopropene ring. Of the two O atoms, the hydroxyl oxygen is nearer to the three-membered ring. This fact may explain the longer distances in the ring in comparison with the compound where the

orientation of the carboxyl group is opposite [Korp, Bernal & Fuchs (1983). *Can. J. Chem.* **61**, 50–56].

Introduction. According to Kobayashi, Arai, Sakuragi, Tokumaru & Utsunomiya (1981), the dihedral angle between the phenyl ring and the double bond in the molecules of *cis*- β -*tert*-butylstyrene is 72°. Thus, sterically bulky *tert*-butyl groups may exert a substantial influence on the position and on the conjugation of phenyl rings with double bonds. The molecule of 2-*tert*-butyl-3-phenyl-2-cyclopropenecarboxylic acid (I) contains the same fragment in a strained three-membered ring. The aim of the present communication is to answer the question whether the *tert*-butyl group will influence the spatial position of the phenyl group in compound (I).

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