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Cyclomicrobuxine monohydrate

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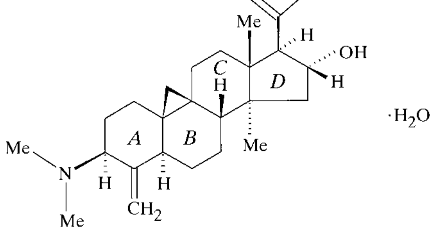
In the title compound, 3β-(dimethylamino)-16α-hydroxy-14-methyl-4-methylene-9,19-cyclo-5α-pregnan-20-one monohydrate, $C_{25}H_{39}NO_2 \cdot H_2O$, the pentacyclo steroidal alkaloid is composed of three six-membered, one five-membered and one three-membered ring. The molecular dimensions are as expected. The structure is stabilized by hydrogen bonds involving H and O atoms of water and the alkaloid molecules, with strong N$\cdots$O [2.829 (7) Å] and O$\cdots$O [2.790 (6) and 2.949 (7) Å] interactions.

Comment

Buxus species have long been known as rich sources of new and biologically active triterpenoidal alkaloids. Previous phytochemical investigations have led to the isolation of 150 such compounds (Atta-ur-Rahman, 1990). The extracts of the genus *Buxus* are reported to be useful in the indigenous system of medicine, in various disorders such as malaria, rheumatism and skin infections (Cordell, 1980). In this article, we report the crystal structure of the title compound, (I), an interesting steroidal alkaloid isolated from the leaves of *B. hyrcana*, collected in Tehran, Iran.



(I)

The crystal structure of (I) is presented in Fig. 1. It contains a pentacyclo steroidal alkaloid hydrogen bonded to a water molecule. The molecular dimensions in (I) are normal and lie within expected ranges for the corresponding bond distances and angles, with mean bond distances as follows: Csp^3-Csp^3 1.535 (7), Csp^3-Csp^2 1.514 (4), $N-Csp^3$ 1.469 (10), C—O 1.430 (6), C=O 1.200 (7) and C=C 1.317 (7) Å. The alkaloid is composed of three six-membered, one five-membered and one three-membered ring. The six-membered rings *A*, *B* and *C* adopt chair, half-chair and skew-boat conformations, respectively, with puckering parameters (Cremer & Pople, 1975) Q = 0.586 (6), 0.530 (6) and 0.638 (5) Å, θ = 3.9 (6), 47.1 (5) and 68.9 (4)° and φ = 263 (9), 91.2 (8) and 273.9 (5)°, respectively. The five-membered ring *D* has a C14-envelope conformation with C14 0.675 (7) Å out of the plane of the remaining four atoms of the ring.

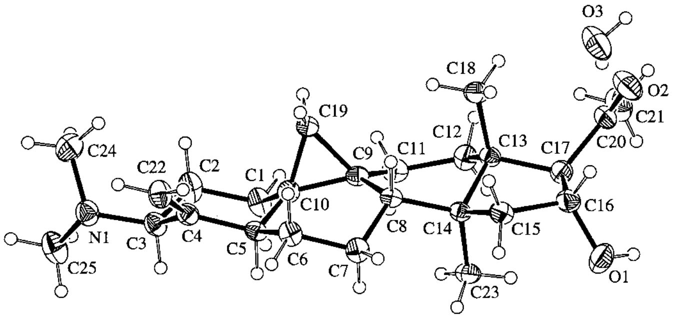


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I) showing the relative stereochemistry and the atomic numbering scheme. Displacement ellipsoids have been plotted at the 30% probability level and H atoms assigned arbitrary radii.

The structure is stabilized by hydrogen bonds involving the alkaloid molecules and water of hydration, with strong N$\cdots$O [2.829 (7) Å] and O$\cdots$O [2.790 (6) and 2.949 (7) Å] interactions. The geometry of the intermolecular interactions is provided in Table 2.

Experimental

The leaves of *Buxus hyrcana* were collected in Tehran, Iran, during the March and April of 1997. The methanolic extract of the air-dried crushed leaves was evaporated to a gum, which was then suspended in water. After defatting with hexane, the aqueous layer was extracted with chloroform at different pH values. The fraction obtained at pH 3 was subjected to column chromatography. Elution of the column with chloroform:methanol (9:1) afforded the crystals of (I). Crystals suitable for X-ray crystallography were grown from acetone:methanol (9:1) at room temperature by slow evaporation.

Crystal data

$C_{25}H_{39}NO_2 \cdot H_2O$
M_r = 403.59
Monoclinic, $P2_1$
a = 6.1333 (8) Å
b = 24.365 (3) Å
c = 8.0681 (6) Å
β = 108.496 (8)°
V = 1143.4 (2) Å^3
Z = 2

D_x = 1.172 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.0–25.0°
μ = 0.075 mm^{-1}
T = 293 (1) K
Prismatic, colourless
0.38 × 0.29 × 0.25 mm

Data collection

Rigaku AFC-6*S* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.972$, $T_{\max} = 0.981$
5848 measured reflections
5257 independent reflections
2901 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$
$\theta_{\max} = 27.55°$
$h = 0 \rightarrow 7$
$k = -31 \rightarrow 31$
$k = -10 \rightarrow 9$
3 standard reflections every 200 reflections intensity decay: 0.12%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.255$
$S = 1.071$
5257 reflections
270 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.1023P)^2 + 1.323P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\max} = -0.24$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.009 (4)

Table 1
Selected geometric parameters (Å, °).

O1—C16	1.430 (6)	C4—C22	1.317 (7)
O2—C20	1.200 (7)	C9—C10	1.525 (7)
N1—C24	1.452 (8)	C9—C19	1.526 (7)
N1—C25	1.473 (8)	C10—C19	1.490 (7)
N1—C3	1.482 (7)		
C24—N1—C25	110.0 (5)	C10—C9—C19	58.5 (3)
C24—N1—C3	114.7 (5)	C19—C10—C9	60.8 (3)
C25—N1—C3	110.6 (5)	C10—C19—C9	60.8 (3)

The space group $P2_1$ was determined from systematic absences, packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure. Friedel opposites (2689) were collected and were not merged. H atoms of the water of solvation and hydroxyl group were located from a difference map. H atoms of the alkaloid were included at riding positions. The water H atoms were left in fixed positions. An absolute configuration could not be established in this analysis. However, refinement of the inverted structure towards the end of the full-matrix least-squares calculations yielded a Flack (1983) parameter of 2 (3) and was, therefore, rejected as the absolute structure present in the crystal.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3—H31$\cdots$O2	0.82	2.17	2.949 (7)	159.4
O3—H32$\cdots$N1$^{\rm i}$	0.82	2.05	2.829 (7)	158.9
O1—H1$\cdots$O3$^{\rm ii}$	0.82	1.98	2.790 (6)	170.6

Symmetry codes: (i) $-x, \frac{1}{2}+y, 1-z$; (ii) $1+x, y, z$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1099). Services for accessing these data are described at the back of the journal.

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