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# Structure of 3β-Dimethylamino-21-norcon-5-enine-20-one Dihydrate

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**Abstract.** $C_{23}H_{36}N_2O.2H_2O$, $M_r = 392{\cdot}6$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}262$ (2), $b = 10{\cdot}127$ (3), $c = 24{\cdot}200$ (3) Å, $Z = 4$, $U = 2269{\cdot}97$ Å$^3$, $D_x = 1{\cdot}149$ Mg m$^{-3}$, $\lambda$(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}6$ mm$^{-1}$, $F(000) = 864$, $T = 293$ K, final $R = 0{\cdot}043$ for 1377 reflections with $I \geq 3\sigma(I)$. Rings *A* and *C* are in the chair conformation. Ring *B* adopts a C(8)β–C(9)α-half-chair conformation. Ring *D* is in a C(14)α-envelope and ring *E* in a C(13)–C(17)-half-chair conformation. The *A*/*B* ring fusion is quasi-*trans*, whilst ring systems *B*/*C* and *C*/*D* are *trans* fused about the bonds C(8)–C(9) and C(13)–C(14), respectively. The *D*/*E* junction shows *cis* fusion.

**Introduction.** The crystal structure of the title compound forms part of a study on steroidal alkaloids (Radhakrishnan, Viswamitra, Bhutani & Ali, 1988). It was isolated from the bark of the plant *Holarrhena antidysenterica* (Bhutani, Ali, Sharma, Vaid & Gupta, 1988). These are important in herbal medicine as antidysentrics and anthelmintics.

**Experimental.** The plate-like crystals were grown from acetone solution by slow evaporation. The cell parameters were refined from accurately determined setting angles of 23 reflections, $9{\cdot}5° < \theta < 30{\cdot}6°$, on a CAD-4 diffractometer. Intensity data were collected with Ni-filtered Cu $K\alpha$ radiation using a crystal of dimensions $0{\cdot}24 \times 0{\cdot}06 \times 0{\cdot}72$ mm up to $(\sin\theta)/\lambda = 0{\cdot}578$ Å$^{-1}$ using $\omega$–$2\theta$ scan. Absorption and extinction corrections were not applied. Lorentz and polarization corrections were applied. A total of 2145 reflections were measured for $0 \leq h \leq 10$, $0 \leq k \leq 11$, $0 \leq l \leq 27$, of which 1377 reflections have $I \geq 3\sigma(I)$. Three strong reflections monitored periodically showed that the crystal was stable to X-rays.

The structure was solved by direct methods using *MULTAN*11/82 (Main *et al.*, 1982). An *E* map computed with the best set of phases (CFOM = 2·746) revealed the positions of 20 out of 28 non-hydrogen atoms. A weighted Fourier synthesis gave the positions of the other eight non-hydrogen atoms. 30 out of 40 H atoms were located by difference synthesis. The positions of 9 of the remaining H atoms were found by geometric considerations. One H atom, attached to O*W*2, was not located. After the final cycle of refinement of the positional parameters of all atoms, the anisotropic temperature factors of non-H atoms and a fixed isotropic temperature factor for H atoms (temperature factors for some H atoms assumed non-positive values when refined) $R = 0{\cdot}043$ and $wR = 0{\cdot}042$. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1$. Function values in the final difference synthesis were between +0·33 and −0·28 e Å$^{-3}$. The maximum shift/e.s.d. for non-H atom parameters was 0·47. All calculations were performed using the Enraf–Nonius (1979) *Structure Determination Package* on a PDP 11/44 computer. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

**Discussion.** The numbering scheme is given in Fig. 1. The packing is shown in Fig. 2. The final coordinates and equivalent isotropic temperature factors for non-hydrogens are given in Table 1.* The bond lengths, bond angles and endocyclic torsion angles are given in Table 2.

Ring *A* is in the chair conformation. The best rotation axis bisecting the bonds C(2)–C(3) and C(5)–C(10) has asymmetry parameter $\Delta C_2$[C(2)–C(3)] = 3·2° (Duax & Norton, 1975). The best mirror passes through C(3) and C(10) with asymmetry parameter $\Delta C_s$[C(3)] = 2·1°. Ring *C* is also in the chair conformation with the best rotational axis bisecting bonds C(9)–C(11) and C(13)–C(14), with $\Delta C_2$-

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51494 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Final fractional coordinates and equivalent isotropic temperature factors for non-hydrogen atoms*

| | *x* | *y* | *z* | $B$(Å$^2$) |
|---|---|---|---|---|
| O(1) | −0·3560 (4) | 0·0932 (4) | 1·0120 (1) | 4·55 (9) |
| O*W*(1) | 0·2950 (5) | 0·1676 (4) | 0·5086 (2) | 5·8 (1) |
| O*W*(2) | 0·4963 (5) | 0·3514 (4) | 0·4679 (2) | 6·3 (1) |
| N(1) | −0·1911 (5) | 0·2135 (4) | 0·9631 (2) | 3·41 (9) |
| N(2) | 0·4533 (5) | 0·0602 (5) | 0·6028 (2) | 4·4 (1) |
| C(1) | 0·3160 (6) | −0·0101 (5) | 0·7504 (2) | 3·6 (1) |
| C(2) | 0·4286 (6) | 0·0182 (6) | 0·7058 (2) | 4·0 (1) |
| C(3) | 0·3551 (6) | 0·0398 (5) | 0·6498 (2) | 3·4 (1) |
| C(4) | 0·2478 (6) | 0·1546 (5) | 0·6552 (2) | 3·8 (1) |
| C(5) | 0·1396 (6) | 0·1290 (5) | 0·7007 (2) | 3·1 (1) |
| C(6) | −0·0003 (6) | 0·1325 (5) | 0·6898 (2) | 3·5 (1) |
| C(7) | −0·1173 (6) | 0·1139 (5) | 0·7312 (2) | 3·5 (1) |
| C(8) | −0·0633 (5) | 0·1196 (5) | 0·7907 (2) | 3·0 (1) |
| C(9) | 0·0802 (5) | 0·0445 (5) | 0·7963 (2) | 3·0 (1) |
| C(10) | 0·1997 (5) | 0·0973 (5) | 0·7575 (2) | 3·1 (1) |
| C(11) | 0·1296 (6) | 0·0323 (6) | 0·8571 (2) | 3·8 (1) |
| C(12) | 0·0118 (6) | −0·0171 (5) | 0·8967 (2) | 3·8 (1) |
| C(13) | −0·1249 (6) | 0·0632 (4) | 0·8909 (2) | 3·0 (1) |
| C(14) | −0·1719 (6) | 0·0633 (5) | 0·8303 (2) | 3·2 (1) |
| C(15) | −0·3270 (6) | 0·1109 (6) | 0·8322 (2) | 3·9 (1) |
| C(16) | −0·3892 (6) | 0·0384 (6) | 0·8820 (2) | 4·3 (1) |
| C(17) | −0·2614 (6) | 0·0175 (5) | 0·9222 (2) | 3·5 (1) |
| C(18) | −0·1014 (6) | 0·2091 (5) | 0·9141 (2) | 3·3 (1) |
| C(19) | 0·2710 (6) | 0·2243 (6) | 0·7813 (2) | 4·3 (1) |
| C(20) | −0·2757 (6) | 0·1090 (5) | 0·9716 (2) | 3·7 (1) |
| C(21) | −0·1872 (6) | 0·3287 (6) | 1·0005 (2) | 4·8 (1) |
| C(22) | 0·5634 (7) | 0·1621 (7) | 0·6106 (3) | 6·3 (2) |
| C(23) | 0·5230 (7) | −0·0644 (6) | 0·5857 (3) | 5·9 (2) |

The equivalent isotropic displacement parameter $B$ is defined as: $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

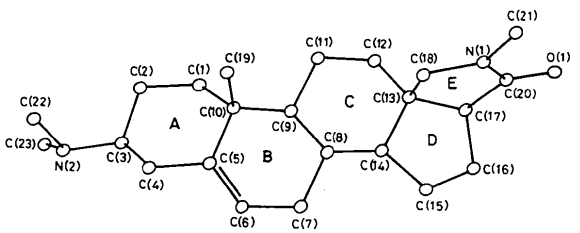


Fig. 1. The numbering scheme.

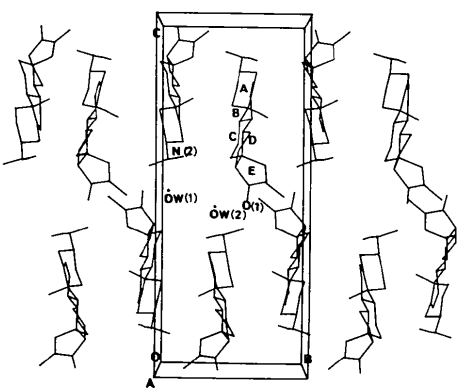


Fig. 2. The packing diagram.

Table 2. *Bond lengths* (Å), *bond angles* (°), *endocyclic torsional angles* (°) *for non-hydrogen atoms and hydrogen bonds*

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| O(1) | C(20) | 1·238 (6) | C(7) | C(8) | 1·525 (7) | | |
| N(1) | C(18) | 1·449 (6) | C(8) | C(9) | 1·537 (7) | | |
| N(1) | C(20) | 1·333 (7) | C(8) | C(14) | 1·503 (7) | | |
| N(1) | C(21) | 1·477 (7) | C(9) | C(10) | 1·547 (7) | | |
| N(2) | C(3) | 1·473 (7) | C(9) | C(11) | 1·545 (7) | | |
| N(2) | C(22) | 1·463 (8) | C(10) | C(19) | 1·556 (7) | | |
| N(2) | C(23) | 1·476 (8) | C(11) | C(12) | 1·537 (7) | | |
| C(1) | C(2) | 1·528 (7) | C(12) | C(13) | 1·512 (7) | | |
| C(1) | C(10) | 1·541 (7) | C(13) | C(14) | 1·531 (7) | | |
| C(2) | C(3) | 1·532 (8) | C(13) | C(17) | 1·546 (7) | | |
| C(3) | C(4) | 1·535 (7) | C(13) | C(18) | 1·594 (7) | | |
| C(4) | C(5) | 1·511 (8) | C(14) | C(15) | 1·516 (8) | | |
| C(5) | C(6) | 1·324 (8) | C(15) | C(16) | 1·525 (8) | | |
| C(5) | C(10) | 1·517 (7) | C(16) | C(17) | 1·548 (8) | | |
| C(6) | C(7) | 1·487 (7) | C(17) | C(20) | 1·518 (8) | | |
| C(18) | N(1) | C(20) | 116·0 (4) | C(1) | C(10) | C(19) | 109·1 (4) |
| C(18) | N(1) | C(21) | 120·9 (4) | C(5) | C(10) | C(9) | 111·1 (4) |
| C(20) | N(1) | C(21) | 123·1 (4) | C(5) | C(10) | C(19) | 108·5 (4) |
| C(3) | N(2) | C(22) | 115·4 (4) | C(9) | C(10) | C(19) | 111·4 (4) |
| C(3) | N(2) | C(23) | 111·4 (4) | C(9) | C(11) | C(12) | 114·2 (4) |
| C(22) | N(2) | C(23) | 109·5 (5) | C(11) | C(12) | C(13) | 111·2 (4) |
| C(2) | C(1) | C(10) | 115·1 (4) | C(12) | C(13) | C(14) | 109·2 (4) |
| C(1) | C(2) | C(3) | 110·4 (4) | C(12) | C(13) | C(17) | 118·7 (5) |
| N(2) | C(3) | C(2) | 115·4 (4) | C(12) | C(13) | C(18) | 110·6 (5) |
| N(2) | C(3) | C(4) | 111·0 (5) | C(14) | C(13) | C(17) | 103·7 (4) |
| C(2) | C(3) | C(4) | 108·7 (5) | C(14) | C(13) | C(18) | 112·1 (4) |
| C(3) | C(4) | C(5) | 111·2 (4) | C(17) | C(13) | C(18) | 102·5 (4) |
| C(4) | C(5) | C(6) | 120·1 (4) | C(8) | C(14) | C(13) | 115·0 (4) |
| C(4) | C(5) | C(10) | 116·9 (4) | C(8) | C(14) | C(15) | 122·2 (4) |
| C(6) | C(5) | C(10) | 123·1 (4) | C(13) | C(14) | C(15) | 104·0 (4) |
| C(5) | C(6) | C(7) | 125·2 (4) | C(14) | C(15) | C(16) | 103·2 (4) |
| C(6) | C(7) | C(8) | 113·0 (4) | C(15) | C(16) | C(17) | 106·0 (4) |
| C(7) | C(8) | C(9) | 110·4 (4) | C(13) | C(17) | C(16) | 106·1 (4) |
| C(7) | C(8) | C(14) | 111·7 (4) | C(13) | C(17) | C(20) | 105·9 (5) |
| C(9) | C(8) | C(14) | 109·5 (4) | C(16) | C(17) | C(20) | 110·2 (4) |
| C(8) | C(9) | C(10) | 113·2 (4) | N(1) | C(18) | C(13) | 103·9 (4) |
| C(8) | C(9) | C(11) | 112·4 (4) | O(1) | C(20) | N(1) | 125·3 (5) |
| C(10) | C(9) | C(11) | 113·3 (5) | O(1) | C(20) | C(17) | 126·5 (5) |
| C(1) | C(10) | C(5) | 107·7 (4) | N(1) | C(20) | C(17) | 108·2 (4) |
| C(1) | C(10) | C(9) | 108·9 (4) | | | | |

Ring torsion angles

Ring *A*

| | |
|---|---|
| 10–1–2–3 | −57·0 (7) |
| 1–2–3–4 | 57·3 (6) |
| 2–3–4–5 | −55·7 (6) |
| 3–4–5–10 | 53·9 (5) |
| 1–10–5–4 | −48·5 (6) |
| 5–10–1–2 | 49·6 (5) |

Ring *B*

| | |
|---|---|
| 6–5–10–9 | 11·2 (6) |
| 10–5–6–7 | 3·4 (7) |
| 5–6–7–8 | 12·7 (8) |
| 6–7–8–9 | −41·8 (6) |
| 7–8–9–10 | 57·4 (6) |
| 8–9–10–5 | −41·4 (6) |

Ring *C*

| | |
|---|---|
| 13–12–11–9 | −51·1 (6) |
| 12–11–9–8 | 49·0 (6) |
| 11–9–8–14 | −49·5 (5) |
| 9–8–14–13 | 55·9 (5) |
| 8–14–13–12 | −58·8 (5) |
| 14–13–12–11 | 53·6 (5) |

Ring *D*

| | |
|---|---|
| 17–13–14–15 | 36·9 (5) |
| 13–14–15–16 | −41·2 (5) |
| 14–15–16–17 | 29·3 (6) |
| 15–16–17–13 | −6·8 (6) |
| 16–17–13–14 | −18·1 (5) |

Ring *E*

| | |
|---|---|
| N(1)–20–17–13 | 15·4 (6) |
| 20–17–13–18 | −18·6 (5) |
| 17–13–18–N(1) | 15·6 (5) |
| 13–18–N(1)–20 | −7·3 (6) |
| 18–N(1)–20–17 | −5·0 (7) |

Hydrogen bonding

| | O···O (Å) | ∠O–H···*X* (°) |
|---|---|---|
| O*W*(1)–H···O*W*(2) | 2·814 | 157·9 |
| O*W*(1)···H–O*W*(2) | 2·832 | 171·9 |
| O*W*(1)–H···N(2) | 2·915 | 159·0 |
| O*W*(2)–H···O(1) | 2·819 | * |

* Not determined.

[C(9)–C(11)] = 1·2°. The best mirror passes through C(11) and C(14), with $\Delta C_s$[C(11)] = 3·1°. Ring *B* is in the C(8)β–C(9)α-half-chair conformation with C(8) and C(9) −0·360 and +0·343 Å away from the plane formed by C(5), C(6), C(7) and C(10). The asymmetry parameter $\Delta C_2$[C(5)–C(6)] = 1·3°.

The large value for the asymmetry parameter {$\Delta C_s$[C(14)] = 8·5°} shows that ring *D* is in a distorted C(14)α-envelope conformation. The phase angle of pseudorotation $\Delta = -47·4°$ and maximum angle of torsion $\varphi = 40·3°$ (Altona, Geise & Romers, 1968). Ring *E*, which has the partial double bond [N(1)–C(20) = 1·33 Å] shows the C(13)–C(17)-half-chair conformation.

The two water molecules are involved in four hydrogen bonds. O*W*(1) forms three H bonds, two with symmetry-related O*W*(2) molecules and one with the N(2) atom. O*W*(2) forms one more H bond with O(1). The torsional angle C(1)–C(10)–C(13)–C(18), which gives the twist of the steroid molecule about the line joining C(10) and C(13), is 50·4°.

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# Structure of (±)-*threo*-9,10,16-Trihydroxypalmitic Acid

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**Abstract.** 9,10,16-Trihydroxyhexadecanoic acid, $C_{16}H_{32}O_5$, $M_r = 304·4$, triclinic, $P\bar{1}$, $a = 4·871$ (1), $b = 8·801$ (1), $c = 21·121$ (2) Å, $\alpha = 89·54$ (1), $\beta = 84·88$ (2), $\gamma = 76·35$ (2)°, $V = 876·14$ (4) Å$^3$, $Z = 2$, $D_x = 1·154$ g cm$^{-3}$, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 0·26$ cm$^{-1}$, $F(000) = 336$, $T = 295$ K. Final $R = 0·055$ for 1556 reflections with $I \geq 3\sigma(I)$. The 16 C atoms form an all-*trans* zigzag chain. Each molecule is linked to seven neighboring molecules *via* eight possible hydrogen bonds with O···O distances varying from 2·591 (6) to 2·673 (3) Å. Each carboxyl group is potentially hydrogen-bonded to the carboxyl and C16–OH groups of other molecules with O···O distances of 2·591 (6) and 2·644 (4) Å. The conformation around the C9–C10 bond is *gauche* with an O3–C9–C10–O4 torsion angle of 66·4 (5)°.

**Introduction.** 9,10,16-Trihydroxypalmitic acid exists as four stereoisomers. Aleuritic acid (I) [(±)-*threo*-9,10,16-trihydroxypalmitic acid], a major constituent of natural shellac (Nagel, 1927), has been studied by various groups (*e.g.* Harries & Nagel, 1922; Ames, Goodburn, Jevans & McGhie, 1968; Eswaran, Seshadri, Sriram & Subramanian, 1971; Chatterjea, Sengupta, Mangee & Mukherjee, 1976). The crystal structure of (I) was originally determined with photographic data by conventional techniques to an $R = 0·11$ (Prasad & Gupta, 1975). It has been redetermined with diffractometer data, the results of which are presented here.

$$HO_2C\text{-}(CH_2)_7\text{-}\underset{OH}{CH}\text{-}\underset{OH}{CH}\text{-}(CH_2)_6\text{-}OH$$

(I)

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