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Delnuttaline, (6β,9β,13β)-13-Acetoxy-6,9-dihydroxyhetisan-2-one

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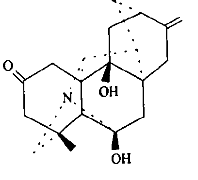
Abstract

The crystal structure of the C-20 diterpenoid alkaloid 6,9-dihydroxy-2-oxohetisan-13-yl acetate consists of two independent molecules in the asymmetric unit with average bond distances $C(sp^3)—C(sp^3)$ 1.55 (1), $C(sp^3)—C(sp^2)$ 1.52 (1), C=C 1.31 (1), N—C 1.51 (1), $C(sp^3)—O$ 1.46 (1), $C(sp^2)—O$ 1.35 (1) and C=O 1.22 (1) Å. The structure is stabilized through a network of hydrogen bonds involving nitrogen and hydroxy groups with O···O and O···N separations in the range 2.688 (7)–2.903 (8) Å.

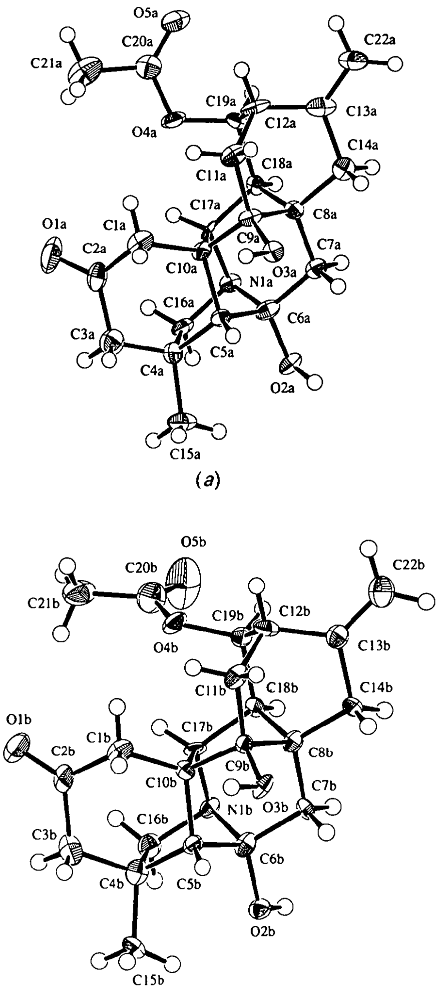
Comment

Delnuttaline (1), a new C-20 diterpenoid alkaloid of the atisine type, was isolated from *Delphinium nuttallianum* Pritz. which is a cow-poisoning plant growing wild in the southern regions of British Columbia, Canada. Suitable crystals of (1) were grown from a mixture of ethanol and ethyl acetate in order to determine its structure and relative configuration by X-ray methods.



(1)

The crystal structure of (1) consists of two independent molecules in the asymmetric unit which differ from each other with respect to the orientation of the acetate groups and of the hydroxy groups at C(6).



(*a*)

(*b*)

Fig. 1. Perspective views of the two independent molecules (*a*) and (*b*) of delnuttaline with crystallographic numbering scheme. Thermal ellipsoids are shown at 50% probability levels; H atoms are assigned an arbitrary radius.

Experimental

Crystal data

$C_{22}H_{27}NO_5$
M_r = 385.46
Monoclinic
$P2_1$
a = 6.623 (3) Å
b = 18.411 (4) Å
c = 15.631 (5) Å
β = 96.75 (2)°

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 19 reflections
θ = 9–15°
μ = 0.095 mm^{-1}
T = 150 (1) K
Block

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$V = 1892.8$ (10) Å^3
$Z = 4$
$D_x = 1.353$ Mg m^{-3}

0.45 × 0.30 × 0.22 mm
Colorless

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (North, Phillips & Mathews, 1968)
$T_{\min} = 0.923$, $T_{\max} = 0.999$
3760 measured reflections
3447 independent reflections
2129 observed reflections [$I > 3.0\sigma(I)$]

$R_{\text{int}} = 0.092$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 21$
$l = -18 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity variation: 2.0% (linear correction)

Refinement

Refinement on F
Final $R = 0.048$
$wR = 0.047$
$S = 2.43$
2129 reflections
504 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\max} = 0.03$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}
Atomic scattering factors from Cromer & Waber (1974) and Stewart, Davidson & Simpson (1965)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{\text{eq}} = (8\pi^2/3)[U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha].$$

	x	y	z	B_{eq}
O(1*a*)	0.1808 (10)	0.5106	−0.1218 (4)	4.5 (4)
O(2*a*)	0.0048 (7)	0.2817 (4)	0.1460 (3)	1.9 (3)
O(3*a*)	0.5405 (7)	0.2441 (4)	0.0096 (3)	1.7 (2)
O(4*a*)	0.0905 (8)	0.3085 (4)	−0.2439 (3)	2.2 (3)
O(5*a*)	0.0525 (9)	0.2622 (4)	−0.3786 (4)	3.0 (3)
N(1*a*)	−0.0744 (9)	0.3176 (4)	0.0014 (4)	1.5 (3)
C(1*a*)	0.3606 (12)	0.3991 (6)	−0.0831 (5)	2.4 (4)
C(2*a*)	0.2481 (13)	0.4670 (6)	−0.0653 (6)	2.5 (4)
C(3*a*)	0.2313 (13)	0.4792 (6)	0.0295 (6)	2.8 (5)
C(4*a*)	0.1431 (12)	0.4137 (5)	0.0731 (5)	1.9 (4)
C(5*a*)	0.2531 (11)	0.3405 (5)	0.0572 (5)	1.4 (4)
C(6*a*)	0.0856 (11)	0.2835 (6)	0.0653 (5)	2.0 (4)
C(7*a*)	0.1368 (12)	0.2067 (5)	0.0387 (5)	1.7 (4)
C(8*a*)	0.2043 (11)	0.2049 (5)	−0.0507 (5)	1.6 (4)
C(9*a*)	0.3790 (12)	0.2582 (5)	−0.0566 (5)	1.8 (4)
C(10*a*)	0.2696 (11)	0.3324 (5)	−0.0427 (5)	1.7 (4)
C(11*a*)	0.4559 (11)	0.2485 (5)	−0.1473 (5)	2.3 (4)
C(12*a*)	0.3033 (13)	0.2031 (6)	−0.2068 (5)	2.4 (4)
C(13*a*)	0.2969 (13)	0.1259 (6)	−0.1717 (5)	2.6 (4)
C(14*a*)	0.2602 (13)	0.1280 (5)	−0.0775 (5)	2.2 (4)
C(15*a*)	0.1562 (13)	0.4339 (6)	0.1702 (5)	2.8 (4)
C(16*a*)	−0.0784 (12)	0.3938 (5)	0.0339 (5)	1.8 (4)
C(17*a*)	0.0391 (11)	0.3177 (5)	−0.0773 (5)	1.3 (3)
C(18*a*)	0.0366 (11)	0.2412 (5)	−0.1137 (5)	1.6 (4)
C(19*a*)	0.0925 (12)	0.2343 (5)	−0.2080 (5)	1.9 (4)
C(20*a*)	0.0812 (12)	0.3139 (6)	−0.3297 (6)	2.4 (4)
C(21*a*)	0.1198 (14)	0.3894 (7)	−0.3554 (6)	3.7 (5)
C(22*a*)	0.3188 (15)	0.0674 (6)	−0.2164 (6)	3.4 (5)
O(1*b*)	−0.4231 (9)	0.2800 (5)	0.6228 (4)	3.5 (3)
O(2*b*)	−0.2978 (8)	0.3310 (4)	0.2382 (3)	1.8 (2)
O(3*b*)	−0.8331 (7)	0.2141 (4)	0.2903 (3)	1.7 (2)
O(4*b*)	−0.3834 (8)	0.0759 (4)	0.4969 (3)	2.2 (3)
O(5*b*)	−0.0745 (10)	0.0249 (5)	0.5129 (5)	6.1 (5)
N(1*b*)	−0.1941 (9)	0.2488 (4)	0.3563 (4)	1.4 (3)
C(1*b*)	−0.6051 (11)	0.2396 (5)	0.4914 (5)	2.0 (4)
C(2*b*)	−0.4792 (12)	0.2917 (5)	0.5475 (5)	2.1 (4)
C(3*b*)	−0.4363 (13)	0.3611 (5)	0.5020 (6)	2.7 (4)
C(4*b*)	−0.3705 (13)	0.3524 (5)	0.4104 (6)	2.2 (4)
C(5*b*)	−0.5054 (11)	0.3007 (5)	0.3531 (5)	1.4 (4)
C(6*b*)	−0.3617 (11)	0.2734 (5)	0.2890 (5)	1.7 (4)
C(7*b*)	−0.4415 (11)	0.2097 (5)	0.2316 (5)	1.5 (4)
C(8*b*)	−0.5161 (11)	0.1466 (5)	0.2827 (5)	1.5 (4)
C(9*b*)	−0.6698 (12)	0.1751 (5)	0.3398 (5)	1.3 (3)
C(10*b*)	−0.5340 (11)	0.2280 (5)	0.4014 (5)	1.4 (4)
C(11*b*)	−0.7607 (11)	0.1102 (5)	0.3847 (5)	1.9 (4)
C(12*b*)	−0.6292 (12)	0.0426 (5)	0.3760 (5)	2.0 (4)
C(13*b*)	−0.6407 (12)	0.0192 (5)	0.2828 (5)	2.0 (4)
C(14*b*)	−0.6017 (12)	0.0845 (5)	0.2266 (5)	1.7 (4)
C(15*b*)	−0.3677 (13)	0.4311 (5)	0.3744 (5)	2.3 (4)
C(16*b*)	−0.1605 (12)	0.3155 (5)	0.4120 (5)	2.1 (4)
C(17*b*)	−0.3120 (12)	0.1968 (5)	0.4046 (5)	1.5 (4)
C(18*b*)	−0.3411 (12)	0.1249 (5)	0.3514 (5)	1.5 (4)
C(19*b*)	−0.4054 (12)	0.0599 (5)	0.4054 (5)	1.8 (4)
C(20*b*)	−0.2052 (15)	0.0569 (6)	0.5428 (6)	3.4 (5)
C(21*b*)	−0.1970 (15)	0.0852 (6)	0.6344 (6)	3.9 (5)
C(22*b*)	−0.6777 (13)	−0.0472 (6)	0.2532 (6)	2.9 (5)

Space group $P2_1$ was determined from systematic absences $0k0$, $k = 2n + 1$. Lorentz-polarization and linear decay corrections were applied to the diffractometer data. The structure was solved by direct methods using *SAPI*91 (Fan, 1991) and refinement was by full-matrix least-squares calculations with non-H atoms anisotropic. H atoms were located from the ΔF map and included at geometrically idealized positions (C—H and O—H 0.95 Å) with fixed isotropic temperature factors. Allowance was made for anomalous dispersion (Ibers & Hamilton, 1964). All calculations were performed using the *TEXSAN* (Molecular Structure Corporation, 1992) crystallographic package installed on a Silicongraphics Personal Iris D/35 computer. Fig. 1 showing the two molecules in the asymmetric unit was drawn using *ORTEP* (Johnson, 1976). A search through the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) yielded only one hit of a closely related compound, cardiopetamine (González, de la Fuente, Reina, Jones & Raithby, 1983).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71192 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1032]

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SHORT COMMUNICATION

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Acta Cryst. (1993). C**49**, 1874

Structures of the 1:1 and 2:1 adducts of (+)-(*S*)-[2.2]paracyclophane-4-carboxylic acid and (–)-ephedrine. Erratum.

By Peter G. Jones and Detlev Döring, *Institut für Anorganische und Analytische Chemie der Technischen Universität, Hagenring* 30, *W*-3300 *Braunschweig, Germany,* and Thomas Laue and Henning Hopf, *Institut für Organische Chemie der Technischen Universität, Hagenring* 30, *W*-3300 *Braunschweig, Germany*

(*Received* 2 *August* 1993)

Abstract

An error in technical editing is corrected. The chemical formulae of the title compounds in the paper by Jones, Döring, Laue & Hopf [*Acta Cryst.* (1993), C**49**, 1192–1195] were reported incorrectly. The correct formulae are $C_{10}H_{16}NO^+.C_{17}H_{15}O_2^-$ and $C_{10}H_{16}NO^+.C_{17}H_{15}O_2^-.C_{17}H_{16}O_2$.

All relevant information is given in the *Abstract.*

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