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1,2,7,8-Tetra-*O*-acetylaustraline

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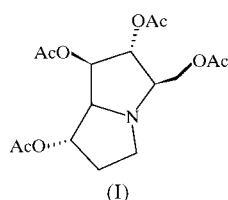
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The title compound, C₁₆H₂₃NO₈, was obtained during studies of australine derivatives. The molecular stereochemistry is similar to that reported for other australine derivatives even though there is no intermolecular hydrogen bonding in this case.

Comment

The title compound, (I), was studied to confirm the stereochemistry as there was some ambiguity between the observed and published NMR spectra. It was isolated as an acylated precursor in the preparation of substituted australine derivatives (Furneaux *et al.*, 1994). The structure consists of independent molecules with normal bond lengths and angles (Allen *et al.*, 1992). There are no close intermolecular contacts and the methyl H atoms on C13 and C17 are disordered over two orientations.



The fused ring C1/C2/C3/N4/C7a is in an envelope conformation with flap atom C2 0.623 (8) Å from the C1/C7a/N4/C3 plane, and with $Q(2) = 0.409$ (5) Å and $\varphi = 247$ (1)° (Cremer & Pople, 1975). This conformation has been found in all previously determined australine structures (Furneaux *et al.*, 1994; Denmark & Herbert, 1998; Nash *et al.*, 1994) except one, namely 1,2,7-tri-*O*-acetyl-8-deoxy-8-fluoro-australine (Furneaux *et al.*, 1994; hereafter WEJVOW), where the twist ring description based on C1–C2 applies [$Q(2) = 0.453$ Å and $\varphi = 239$ °]. The fused ring N4/C5/C6/C7/C7a adopts the twist-ring conformation based on C5–C6, with $Q(2) = 0.394$ (6) Å and $\varphi = 82.1$ (8)°, very similar to that found in WEJVOW [$Q(2) = 0.414$ Å and $\varphi = 83$ °]. We note the close stereochemical similarity with the published structures which indicates that for

australine derivatives the molecular geometry is not perturbed by intermolecular hydrogen-bonding crystal-packing forces; for example, in casaurine (Nash *et al.*, 1994), all five hydroxyl protons are involved in O–H...O hydrogen bonds.

Experimental

Crystals of 1,2,7,8-tetra-*O*-acetylaustraline were obtained as a minor component following acetylation and chromatographic fractionation of the mother liquor remaining after crystallizing castanospermine from extracts of *Castanospermine australe* seeds.

Crystal data

C ₁₆ H ₂₃ NO ₈	$D_x = 1.367$ Mg m ⁻³
$M_r = 357.35$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 25 reflections
$a = 9.586$ (3) Å	$\theta = 4.9$ – 14.2 °
$b = 8.279$ (2) Å	$\mu = 0.110$ mm ⁻¹
$c = 11.096$ (5) Å	$T = 173$ (2) K
$\beta = 99.74$ (2)°	Translucent, colourless
$V = 867.9$ (5) Å ³	$0.44 \times 0.26 \times 0.16$ mm
$Z = 2$	

Data collection

Siemens R3m diffractometer	$h = 0 \rightarrow 11$
ω scans	$k = 0 \rightarrow 9$
1794 measured reflections	$l = -13 \rightarrow 13$
1648 independent reflections	3 standard reflections
1190 reflections with $I > 2\sigma(I)$	every 97 reflections
$R_{int} = 0.026$	intensity decay: none
$\theta_{max} = 25$ °	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.053$	$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2]$
$wR(F^2) = 0.108$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.012$	$(\Delta/\sigma)_{max} < 0.001$
1648 reflections	$\Delta\rho_{max} = 0.22$ e Å ⁻³
228 parameters	$\Delta\rho_{min} = -0.27$ e Å ⁻³

Table 1

Selected geometric parameters (Å, °).

O1–C10	1.357 (7)	C1–C7a	1.541 (7)
O1–C1	1.431 (5)	C2–C3	1.527 (7)
N4–C3	1.458 (6)	C3–C8	1.509 (7)
N4–C5	1.461 (6)	C5–C6	1.533 (7)
N4–C7a	1.494 (6)	C6–C7	1.527 (7)
C1–C2	1.515 (7)	C7–C7a	1.525 (7)
C10–O1–C1	115.7 (4)	N4–C3–C2	102.2 (4)
C3–N4–C5	116.6 (4)	C8–C3–C2	115.6 (4)
C3–N4–C7a	109.2 (4)	N4–C5–C6	105.8 (4)
C5–N4–C7a	108.2 (4)	C7–C6–C5	101.2 (4)
O1–C1–C2	109.8 (4)	N4–C7a–C7	105.4 (4)
O1–C1–C7a	114.6 (4)	C7–C7a–C1	118.7 (5)
C2–C1–C7a	102.8 (4)	O2–C10–O1	123.8 (5)
C1–C2–C3	102.9 (4)	O2–C10–C11	126.2 (5)
N4–C3–C8	114.4 (4)	O1–C10–C11	110.0 (5)
C10–O1–C1–C2	168.1 (4)	C7a–N4–C5–C6	–18.4 (6)
C12–O3–C2–C1	112.7 (5)	N4–C5–C6–C7	35.9 (5)
C7a–C1–C2–C3	38.5 (5)	C5–C6–C7–C7a	–39.3 (5)
C5–N4–C3–C2	151.9 (4)	C3–N4–C7a–C7	121.0 (4)
C7a–N4–C3–C2	29.0 (5)	C5–N4–C7a–C7	–6.8 (5)
C1–C2–C3–N4	–41.8 (5)	C2–C1–C7a–N4	–21.0 (5)
C3–N4–C5–C6	–141.9 (4)	C2–C1–C7a–C7	–138.4 (4)

All H atoms were constrained to an isotropic displacement parameter 1.2 times that of the U_{eq} value of their parent atom. No Friedel pairs were collected. The absolute configuration cannot be deter-

mined from this experiment and was assumed from the synthesis.

Data collection: *R3m Software* (Siemens, 1983); cell refinement: *R3m Software*; data reduction: *R3m Software*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97*.

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