C2—C3	1.537 (6)	C8—C9	1.531 (5)
C2—C7	1.511 (4)	C8—C10	1.527 (6)
C2—C8	1.553 (5)	N1—H1*a*	0.89 (4)
C2—C11	1.502 (5)		
N3—N2—C1	118.5 (3)	C4—C5—C6	106.3 (4)
N2—N3—C7	116.8 (3)	C4—C5—C8	102.6 (4)
O1—C1—N1	122.9 (3)	C6—C5—C8	103.3 (3)
O1—C1—N2	121.0 (3)	C5—C6—C7	101.5 (3)
N1—C1—N2	116.1 (3)	N3—C7—C2	123.1 (3)
C3—C2—C7	104.5 (3)	N3—C7—C6	129.5 (3)
C3—C2—C8	101.8 (3)	C2—C7—C6	107.3 (3)
C3—C2—C11	113.8 (3)	C2—C8—C5	93.8 (3)
C7—C2—C8	99.3 (3)	C2—C8—C9	114.1 (4)
C7—C2—C11	116.0 (3)	C2—C8—C10	112.7 (3)
C8—C2—C11	119.2 (3)	C5—C8—C9	113.8 (3)
C2—C3—C4	103.8 (4)	C5—C8—C10	114.4 (4)
C3—C4—C5	103.1 (4)	C9—C8—C10	107.8 (4)

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
$N1—H1b\cdots O1^{iii}$	0.90 (4)	2.05 (4)	2.933 (4)	167 (4)
$N2—H2\cdots O1^{i}$	0.83 (4)	2.31 (4)	3.137 (4)	177 (4)

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

The scan rate was 6° min^{-1} (in ω). Weak reflections [$I < 10\sigma(I)$] were rescanned (maximum of three rescans) and the counts accumulated to improve accuracy. The positional parameters of atoms H1*a*, H1*b* and H2 were refined; all other H atoms were placed at assumed positions (C,N—H = 0.95 Å, $U = 1.2 \times U_{eq}$ of associated C atom) and fixed. Data Collection *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *TEXSAN*, *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *TEXSAN FINISH*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71746 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1088]

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Acta Cryst. (1994). **C50**, 734–736

3-β-D-Ribofuranosyl-6,7-dihydro-9*H*-thiazolo[3,2-*a*]purin-9-one Hydrate

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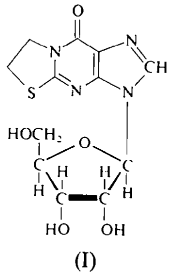
Abstract

The title molecule, $C_{12}H_{14}N_4O_5S.H_2O$ (I), has a *syn*-χ_{CN} glycosyl torsion angle, which is stabilized by an intramolecular hydrogen bond between N3 of the tricylic base and O5′ of the ribose (in a C2′-*endo* pucker). [The purine base, including atoms S and O6, of the molecule is planar to within 0.043 (2) Å.] The tricyclic bases are stacked along *a* with an interplanar distance of 3.602 (3) Å.

Comment

As part of anti-tumour drug research, the structure of (I) has been determined in order to elucidate the sugar pucker and glycosyl torsion conformations. Details of the synthetic work have been published elsewhere (Chern, Lin & Chen, 1990).



(I)

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The crystallographic analysis shows that the ribose ring adopts a C2′-*endo* pucker conformation and that the intra-annular torsion angles starting from C1′ and proceeding around the C1′—C2′—C3′—C4′—O4′ ring are −32.9, 18.1, 6.0, −27.7 and 37.8 (3)°, respectively. Interestingly, the *syn*-glycosyl conformation is stabilized by an intramolecular hydrogen bond [2.18 (4) Å] between N3 of the tricyclic base and O5′ of the ribose. While the '*gauche* effect' (Wolfe, 1972) suggested that ribose sugar favours a C3′-*endo* pucker conformation, our results clearly indicate that other puckers are equally likely. Three hydrogen bonds from water and one between two molecules stabilize the lattice packing.

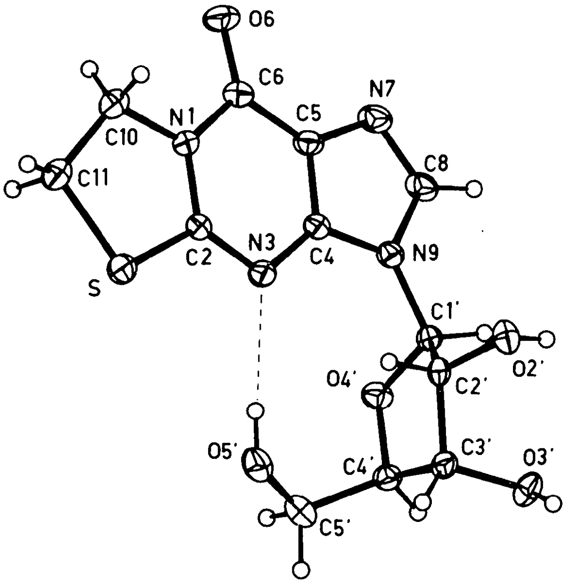

Fig. 1. Structure of (I) showing 50% probability displacement ellipsoids for the non-H atoms; H atoms are drawn as small spheres of arbitrary radii.

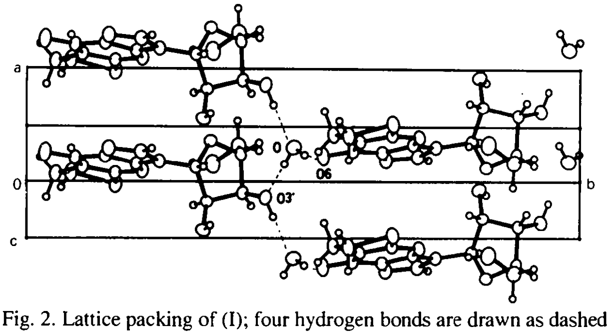

Fig. 2. Lattice packing of (I); four hydrogen bonds are drawn as dashed lines.

Experimental

Crystal data

$C_{12}H_{14}N_4O_5S.H_2O$
M_r = 344.34
Monoclinic
$P2_1$
a = 4.9635 (4) Å
b = 21.935 (2) Å
c = 6.7924 (5) Å
β = 101.096 (6)°
V = 725.68 (10) Å^3
Z = 2
D_x = 1.576 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 24 reflections
θ = 25–40°
μ = 2.363 mm^{-1}
T = 293 (2) K
Plate
0.4 × 0.35 × 0.2 mm
Colorless

Data collection

Rigaku AFC-5*R*/RU300 diffractometer
$\omega/2\theta$ scans [width (1.0 + 0.14tanθ)° in ω; rate 4° min^{-1}]
Absorption correction: empirical
T_{min} = 0.92, T_{max} = 0.95
1253 measured reflections
1114 independent reflections

1102 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0132
θ_{max} = 60.01°
$h = 0 \rightarrow 5$
$k = 0 \rightarrow 24$
$l = -7 \rightarrow 7$
3 standard reflections
frequency: 150 min
intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0222
$wR(F^2)$ = 0.0597
S = 1.078
1114 reflections
273 parameters
All H-atom parameters refined
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 0.1009P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.059

$\Delta\rho_{max}$ = 0.169 e Å^{-3}
$\Delta\rho_{min}$ = −0.149 e Å^{-3}
Extinction correction: $F_c^* = kF_c[1 + (0.001\chi \times F_c^2\lambda^3/\sin2\theta)]^{-1/4}$
Extinction coefficient: χ = 0.0081 (10)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
S	0.85196 (15)	0.65872 (3)	0.23980 (10)	0.0389 (2)
N1	1.1254 (5)	0.59994 (10)	0.0108 (4)	0.0308 (5)
C2	1.0496 (5)	0.65745 (14)	0.0539 (4)	0.0291 (6)
N3	1.1093 (5)	0.70757 (11)	−0.0337 (4)	0.0317 (5)
C4	1.2524 (5)	0.69581 (13)	−0.1813 (4)	0.0284 (6)
C5	1.3471 (5)	0.63969 (13)	−0.2328 (4)	0.0321 (6)
C6	1.2819 (6)	0.58606 (14)	−0.1334 (4)	0.0345 (6)
O6	1.3423 (5)	0.53263 (10)	−0.1608 (3)	0.0487 (6)
N7	1.4933 (5)	0.64608 (12)	−0.3854 (4)	0.0393 (6)
C8	1.4826 (6)	0.70456 (15)	−0.4239 (4)	0.0357 (7)
N9	1.3378 (4)	0.73700 (11)	−0.3056 (3)	0.0305 (5)
C10	1.0104 (8)	0.55108 (15)	0.1183 (5)	0.0396 (7)
C11	0.9275 (8)	0.5788 (2)	0.2989 (5)	0.0452 (8)
C1′	1.2927 (5)	0.80261 (13)	−0.3169 (4)	0.0272 (6)
C2′	0.9935 (5)	0.82235 (13)	−0.3328 (4)	0.0262 (6)
O2′	0.8287 (4)	0.81575 (10)	−0.5246 (3)	0.0355 (5)
C3′	1.0289 (5)	0.88725 (13)	−0.2526 (4)	0.0296 (6)
O3′	1.0669 (5)	0.92870 (10)	−0.4063 (3)	0.0391 (5)
C4′	1.2956 (6)	0.88370 (13)	−0.0947 (4)	0.0314 (6)
O4′	1.4349 (4)	0.82882 (9)	−0.1376 (3)	0.0337 (5)
C5′	1.2592 (7)	0.8800 (2)	0.1203 (5)	0.0437 (8)
O5′	1.0797 (5)	0.83291 (13)	0.1536 (3)	0.0483 (6)
O	0.5568 (5)	0.97957 (12)	0.5118 (4)	0.0463 (6)

Table 2. *Selected geometric parameters* (Å, °)

S—C2	1.742 (3)	C8—N9	1.375 (4)
S—C11	1.821 (3)	N9—C1′	1.456 (4)
N1—C2	1.364 (4)	C10—C11	1.496 (5)
N1—C6	1.396 (4)	C1′—O4′	1.407 (3)
N1—C10	1.473 (4)	C1′—C2′	1.531 (3)
C2—N3	1.311 (4)	C2′—O2′	1.406 (3)
N3—C4	1.360 (4)	C2′—C3′	1.522 (4)
C4—N9	1.359 (4)	C3′—O3′	1.424 (3)
C4—C5	1.386 (4)	C3′—C4′	1.537 (4)
C5—N7	1.382 (4)	C4′—O4′	1.446 (4)
C5—C6	1.424 (4)	C4′—C5′	1.508 (4)
C6—O6	1.233 (4)	C5′—O5′	1.410 (4)
N7—C8	1.308 (4)		
C2—S—C11	91.36 (15)	C4—N9—C8	106.2 (2)
C2—N1—C6	124.5 (2)	C4—N9—C1′	128.8 (2)
C2—N1—C10	114.6 (2)	C8—N9—C1′	125.0 (2)
C6—N1—C10	120.7 (2)	N1—C10—C11	107.7 (3)
N3—C2—N1	125.6 (2)	C10—C11—S	106.7 (2)
N3—C2—S	121.6 (2)	O4′—C1′—N9	108.2 (2)
N1—C2—S	112.8 (2)	O4′—C1′—C2′	105.3 (2)
C2—N3—C4	111.8 (2)	N9—C1′—C2′	114.9 (2)
N9—C4—N3	127.0 (3)	O2′—C2′—C3′	115.7 (2)
N9—C4—C5	105.8 (2)	O2′—C2′—C1′	114.8 (2)
N3—C4—C5	127.2 (3)	C3′—C2′—C1′	101.3 (2)
N7—C5—C4	110.6 (2)	O3′—C3′—C2′	110.9 (2)
N7—C5—C6	129.7 (3)	O3′—C3′—C4′	109.0 (2)
C4—C5—C6	119.7 (2)	C2′—C3′—C4′	103.1 (2)
O6—C6—N1	119.8 (3)	O4′—C4′—C5′	107.6 (2)
O6—C6—C5	129.0 (3)	O4′—C4′—C3′	106.4 (2)
N1—C6—C5	111.1 (3)	C5′—C4′—C3′	115.5 (3)
C8—N7—C5	104.0 (2)	C1′—O4′—C4′	109.6 (2)
N7—C8—N9	113.3 (3)	O5′—C5′—C4′	113.1 (3)

Table 3. *Contact distances* (Å)

A	H	*D*	*A*···H	*D*···*A*
N3	H5O′	O5′	2.21 (6)	3.045 (4)
O^{i}	H3O′	O3′	1.93 (4)	2.725 (4)
O6	H1	O	2.03 (4)	2.784 (3)
O3′^{ii}	H2	O3	1.98 (6)	2.826 (4)
O5′^{i}	H2O′	O2′	1.98 (4)	2.742 (3)

Symmetry codes: (i) $x, y, 1 - z$; (ii) $1 - x, y, 1 + z$.

Dispersion corrections and absorption coefficients were taken from *International Tables for Crystallography* (1992, Vol. C, Tables 6.1.1.4, 4.2.6.8 and 4.2.4.2). Since (I) crystallizes in a polar space group, polar-axis restraints were applied by the method of Flack & Schwarzenbach (1988) and the absolute structure of the crystal was established as described by Flack (1983), which is consistent with the known absolute configuration of β-D-ribofuranose. Data collection and cell refinement: Rigaku AFC-5 software. Data reduction: *NRCVAX* (Gabe, Lee & Le Page, 1985). Program used to solve structure: *SHELXS86* (Sheldrick, 1990). Program used to refine structure: *SHELXL* (Sheldrick, 1994). Molecular graphics: *ORTEP* (Johnson, 1971). Software used to prepare material for publication: *SHELXL*; *NRCVAX*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71785 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1058]

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Absolute Stereostructure of 13,17-Epoxyalisol B 23-Acetate Isolated from *Alisma orientale*

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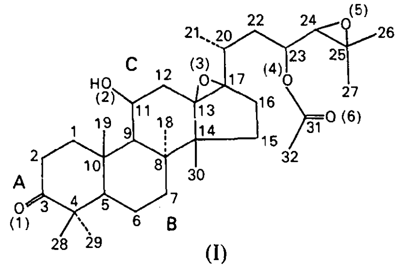
Abstract

The absolute configration of the epoxy group in 13β,17β:-24(*R*),25-diepoxy-1β-hydroxydammar-3-one 23(*S*)-acetate, $C_{32}H_{50}O_6$, m.p. 472-474 K, $[\alpha]_D$+139.4° (*c* = 0.96, $CHCl_3$), isolated from *Alisma orientale* rhizomes, was established to be the β orientation by X-ray crystallographic analysis. The *A*, *B* and *C* rings have chair forms. An intermolecular hydrogen bond is observed between O2 and O6′ with a distance of 3.148 (6) Å.

Comment

13,17-Epoxyalisol B 23-acetate (I) was isolated from *Alisma orientale* rhizomes along with 20 related triterpenes of the protostane type (Nakajima, Mikoshiba, Ida & Shoji, 1984; Ida, Satoh, Nakajima, Yamaguchi & Shoji, 1989).



(I)