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Structure of (4*S**,6*R**)-(8-Oxo-3,9-dioxa-1-aza-4-bicyclo[4.3.0]nonanyl)methyl Acetate

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Abstract. $C_9H_{13}NO_5$, $M_r = 215{\cdot}2$, monoclinic, $C2/c$, $a = 27{\cdot}192\ (4)$, $b = 5{\cdot}714\ (2)$, $c = 15{\cdot}257\ (3)$ Å, $\beta = 119{\cdot}57\ (2)°$, $V = 2062\ (1)$ Å^3, $Z = 8$, $D_x = 1{\cdot}387$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 1·067 cm^{-1}, $F(000) = 912$, $T = 298$ K, final $R = 0{\cdot}047$ for 1186 reflections. The six-membered ring is in a chair conformation and the five-membered ring has a half-chair conformation. The least-squares planes, defined by the atoms of the two rings, form a dihedral angle of 111·7 (1)°. The shortest non-bonded contact is 3·16 Å for the non-H atoms.

Experimental. Crystal of approximate dimensions 0·21 × 0·35 × 0·56 mm; intensities measured at 298 K on an Enraf–Nonius CAD-4 four-circle diffractometer (Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71073$ Å, graphite monochromator). Lattice parameters determined by least squares from 20 reflections ($10 < 2\theta < 24°$). Total of 2059 reflections up to $\theta = 25°$ ($-32 \le h \le 26$, $0 \le k \le 6$, $0 \le l \le 18$) measured in the ω–2θ scan mode, 1756 unique reflections ($R_{int} = 0{\cdot}013$), 1186 reflections considered as observed [$F_o > 2\sigma(F_o)$]. The intensity variation of three standard reflections, measured every hour, was less than 2%. No absorption or secondary-extinction corrections. Structure solved by *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least squares with *SDP* (B. A. Frenz & Associates Inc., 1985). Weights of each reflection in refinement (on F) calculated from $w = 1/\sigma^2(F_o)$, $\sigma(F_o)$ being the e.s.d., based on counting statistics, of the observed structure factor. Scattering factors taken from *International Tables for X-ray Crystallography* (1974). All the H atoms included in the refinement in calculated positions [d(C—H) = 0·95 Å]. The total number of parameters refined was

Table 1. *Fractional atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2) *for non-*H *atoms with e.s.d.'s in parentheses*

$B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	B_{eq}
N(1)	1905·5 (7)	10902 (4)	9112 (1)	3·18 (5)
C(2)	1526 (1)	9030 (5)	9019 (2)	3·80 (7)
O(3)	1293·1 (7)	7755 (3)	8111 (1)	3·36 (4)
C(4)	982·4 (9)	9228 (5)	7260 (2)	3·15 (6)
C(5)	1384·7 (9)	11017 (5)	7219 (2)	3·50 (7)
C(6)	1697 (1)	12377 (5)	8202 (2)	3·41 (7)
C(7)	2249 (1)	13392 (5)	8365 (2)	3·99 (7)
C(8)	2645 (1)	11379 (6)	8830 (2)	3·78 (7)
O(8)	3099·9 (7)	11032 (5)	8912 (1)	5·78 (6)
O(9)	2416·2 (6)	9814 (3)	9183 (1)	3·49 (4)
C(10)	733 (1)	7707 (5)	6335 (2)	3·77 (7)
O(10)	362·3 (6)	5927 (3)	6361 (1)	3·82 (5)
C(11)	−180 (1)	6490 (5)	6004 (2)	3·72 (7)
O(11)	−354·0 (8)	8423 (4)	5700 (2)	6·79 (7)
C(12)	−517 (1)	4543 (5)	6066 (2)	4·49 (8)

Table 2. *Bond distances* (Å), *bond angles* (°) *and torsion angles* (°) *for non-*H *atoms with e.s.d.'s in parentheses*

N(1)—C(2)	1·444 (4)	C(6)—C(7)	1·511 (5)
N(1)—C(6)	1·478 (3)	C(7)—C(8)	1·495 (4)
N(1)—O(9)	1·476 (3)	C(8)—O(8)	1·197 (4)
C(2)—O(3)	1·408 (3)	C(8)—O(9)	1·344 (4)
O(3)—C(4)	1·424 (3)	C(10)—O(10)	1·446 (4)
C(4)—C(5)	1·521 (4)	O(10)—C(11)	1·335 (4)
C(4)—C(10)	1·504 (3)	C(11)—O(11)	1·201 (5)
C(5)—C(6)	1·522 (3)	C(11)—C(12)	1·474 (4)

C(2)—N(1)—C(6)	113·9 (2)	C(5)—C(6)—C(7)	112·7 (3)
C(2)—N(1)—O(9)	107·3 (2)	C(6)—C(7)—C(8)	102·0 (2)
C(6)—N(1)—O(9)	102·7 (3)	C(7)—C(8)—O(8)	130·0 (4)
N(1)—C(2)—O(3)	115·6 (2)	C(7)—C(8)—O(9)	109·5 (2)
C(2)—O(3)—C(4)	111·6 (2)	O(8)—C(8)—O(9)	120·5 (3)
O(3)—C(4)—C(5)	108·7 (2)	N(1)—O(9)—C(8)	107·2 (2)
O(3)—C(4)—C(10)	107·7 (3)	C(4)—C(10)—O(10)	112·3 (2)
C(5)—C(4)—C(10)	110·5 (2)	C(10)—O(10)—C(11)	117·6 (3)
C(4)—C(5)—C(6)	111·4 (2)	O(10)—C(11)—O(11)	121·6 (3)
N(1)—C(6)—C(5)	113·9 (3)	O(10)—C(11)—C(12)	112·9 (2)
N(1)—C(6)—C(7)	100·1 (2)	O(11)—C(11)—C(12)	125·4 (3)

C(6)—N(1)—C(2)—O(3)	45·1 (3)	O(3)—C(4)—C(10)—O(10)	59·4 (3)
O(9)—N(1)—C(2)—O(3)	−67·8 (3)	C(5)—C(4)—C(10)—O(10)	178·0 (2)
C(2)—N(1)—C(6)—C(5)	−37·6 (3)	C(4)—C(5)—C(6)—N(1)	43·4 (3)
C(2)—N(1)—C(6)—C(7)	−157·8 (2)	C(4)—C(5)—C(6)—C(7)	156·4 (2)
O(9)—N(1)—C(6)—C(5)	78·1 (3)	N(1)—C(6)—C(7)—C(8)	36·5 (2)
O(9)—N(1)—C(6)—C(7)	−42·2 (2)	C(5)—C(6)—C(7)—C(8)	−84·7 (2)
C(2)—N(1)—O(9)—C(8)	153·4 (2)	C(6)—C(7)—C(8)—O(8)	161·7 (3)
C(6)—N(1)—O(9)—C(8)	33·0 (2)	C(6)—C(7)—C(8)—O(8)	−17·8 (3)
N(1)—C(2)—O(3)—C(4)	−58·9 (3)	C(7)—C(8)—O(9)—N(1)	−9·0 (2)
C(2)—O(3)—C(4)—C(5)	62·4 (3)	O(8)—C(8)—O(9)—N(1)	171·4 (2)
C(2)—O(3)—C(4)—C(10)	−177·8 (2)	C(4)—C(10)—O(10)—C(11)	87·6 (3)
O(3)—C(4)—C(5)—C(6)	−55·0 (3)	C(10)—O(10)—C(11)—O(11)	−2·4 (4)
C(10)—C(4)—C(5)—C(6)	−173·0 (2)	C(10)—O(10)—C(11)—C(12)	179·8 (2)

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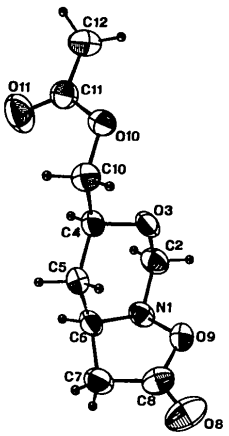

Fig. 1. *ORTEP*II (Johnson, 1976) view of the molecule with the atom numbering. The thermal ellipsoids are given at 50% probability.

149: one scale factor, position parameters and anisotropic thermal parameters for non-H atoms, and isotropic thermal parameters for H atoms. Refinement resulted in final values of $R = 0{\cdot}047$, $wR = 0{\cdot}041$ and $S = 2{\cdot}60$; in the last cycle $(\Delta/\sigma)_{max} = 0{\cdot}04$; final max. and min. $\Delta\rho$ 0·22 and $-0{\cdot}24$ e Å^{-3}, respectively. All calculations performed on a Micro-PDP 11/73 computer. The final atomic coordinates are given in Table 1.* Bond distances, bond angles and torsion angles are given in Table 2. The atom numbering is shown in Fig. 1 and unit-cell contents in Fig. 2.

* Lists of structure factors, anisotropic thermal parameters for the non-H atoms, positional and isotropic thermal parameters for the H atoms, and shortest non-bonded contacts have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52027 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

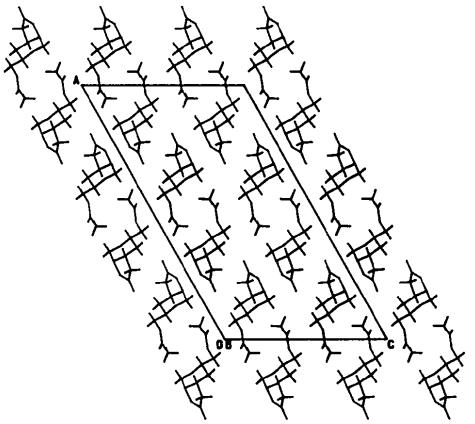

Fig. 2. Packing diagram.

Related literature. The organic synthesis and spectroscopic data for the title compound (Panfil, Bełżecki, Chmielewski & Suwińska, 1989) have been published.

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