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Structure of (3*R*,6*R*,7*R*,11*S*)-7-Acetoxymethyl-5,8,10-trioxa-1-azatricyclo[4.3.2.$0^{3,11}$]undecan-4-one

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Abstract. $C_{10}H_{13}NO_6$, $M_r = 243{\cdot}2$, monoclinic, $P2_1$, $a = 5{\cdot}558\ (2)$, $b = 8{\cdot}119\ (4)$, $c = 11{\cdot}947\ (4)$ Å, $\beta = 98{\cdot}19\ (2)°$, $V = 534\ (2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}514$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}185$ cm^{-1}, $F(000) = 256$, $T = 298$ K, final $R = 0{\cdot}046$ for 920 observed reflections. The two five-membered rings adopt different conformations: envelope and half-chair. The seven-membered ring shows the distorted chair conformation. The shortest non-bonded contact is 3·29 Å for non-H atoms.

Experimental. Crystal of approximate dimensions 0·28 × 0·28 × 0·30 mm; intensities measured at 298 K on an Enraf–Nonius CAD-4 four-circle diffractometer (Mo $K\alpha$ radiation, graphite monochromator). Lattice parameters determined by least squares from 20 reflections ($12 \le 2\theta \le 28°$). Total of 1174 reflections up to $\theta = 26°$ ($0 \le h \le 6$, $0 \le k \le 10$, $-14 \le l \le 14$) measured in the ω–2θ scan mode, 920 reflections considered as observed [$F_o > 1{\cdot}5\sigma(F_o)$]. Three reference reflections monitored every hour showed no significant variation in intensity. No absorption or secondary-extinction correction. Structure solved by *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least squares with the *SDP* system (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). The total number of parameters refined was 153: one scale factor, position parameters and anisotropic thermal parameters for non-H atoms; no attempt was made to refine the positions [calculated at d(C—H) = 0·95 Å] or isotropic thermal parameters ($B = 5{\cdot}0$ Å^2) of the H atoms. Refinement resulted in final values of $R = 0{\cdot}046$, $wR = 0{\cdot}049$ and $S = 3{\cdot}18$; in the last cycle $(\Delta/\sigma)_{max} = 0{\cdot}02$. Final max. and min. $\Delta\rho$ were 0·21 and $-0{\cdot}28$ e Å^{-3}, respectively. All calculations performed on a MicroPDP11/73 computer. The final

Table 1. *Fractional atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2) *for non-*H *atoms*

E.s.d.'s are 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}
C(1)	6743 (7)	9188 (6)	3037 (3)	2·85 (8)
O(2)	8225 (5)	7740*	2927 (2)	3·35 (6)
C(3)	8373 (8)	7438 (6)	1829 (4)	3·57 (9)
O(3)	9739 (7)	6432 (5)	1555 (3)	6·46 (9)
C(4)	6521 (8)	8443 (6)	1072 (3)	3·62 (9)
C(5)	7581 (8)	9748 (7)	362 (3)	4·1 (1)
N(6)	7212 (6)	11351 (5)	881 (3)	3·56 (7)
C(7)	9187 (8)	11937 (6)	1696 (3)	3·70 (9)
O(8)	10055 (4)	10827 (4)	2582 (2)	2·71 (5)
C(9)	8402 (7)	10615 (5)	3399 (3)	2·48 (7)
C(10)	5205 (7)	9435 (6)	1868 (3)	3·34 (9)
O(11)	5115 (5)	11094 (4)	1457 (2)	3·73 (6)
C(12)	10061 (7)	10343 (6)	4516 (3)	2·89 (8)
O(12)	8813 (5)	9580 (4)	5371 (2)	2·83 (5)
C(13)	7069 (7)	10491 (5)	5764 (3)	2·83 (8)
O(13)	6515 (5)	11839 (4)	5423 (2)	3·66 (6)
C(14)	5975 (8)	9573 (7)	6648 (3)	3·74 (9)

* Origin-defining coordinate.

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Table 2. *Bond distances* (Å), *bond angles* (°) *and torsion angles* (°) *for non-*H *atoms*

E.s.d.'s are in parentheses.

C(1)—O(2)	1·451 (5)	N(6)—O(11)	1·450 (4)
C(1)—C(9)	1·506 (6)	C(7)—O(8)	1·421 (5)
C(1)—C(10)	1·543 (5)	O(8)—C(9)	1·443 (5)
O(2)—C(3)	1·348 (6)	C(9)—C(12)	1·527 (5)
C(3)—O(3)	1·193 (6)	C(10)—O(11)	1·432 (7)
C(3)—C(4)	1·509 (6)	C(12)—O(12)	1·452 (5)
C(4)—C(5)	1·528 (8)	O(12)—C(13)	1·355 (5)
C(4)—C(10)	1·512 (6)	C(13)—O(13)	1·194 (5)
C(5)—N(6)	1·469 (7)	C(13)—C(14)	1·491 (6)
N(6)—C(7)	1·441 (5)		

O(2)—C(1)—C(9)	108·4 (3)	N(6)—C(7)—O(8)	116·0 (4)
O(2)—C(1)—C(10)	105·8 (3)	C(7)—O(8)—C(9)	113·4 (3)
C(9)—C(1)—C(10)	113·4 (4)	C(1)—C(9)—O(8)	108·6 (4)
C(1)—O(2)—C(3)	110·5 (3)	C(1)—C(9)—C(12)	114·2 (3)
O(2)—C(3)—O(3)	121·5 (4)	O(8)—C(9)—C(12)	104·2 (3)
O(2)—C(3)—C(4)	110·7 (4)	C(1)—C(10)—C(4)	104·2 (3)
O(3)—C(3)—C(4)	127·7 (5)	C(1)—C(10)—O(11)	114·9 (4)
C(3)—C(4)—C(5)	115·1 (4)	C(4)—C(10)—O(11)	106·5 (3)
C(3)—C(4)—C(10)	105·1 (3)	N(6)—O(11)—C(10)	107·9 (4)
C(5)—C(4)—C(10)	103·8 (5)	C(9)—C(12)—O(12)	112·7 (3)
C(4)—C(5)—N(6)	107·0 (4)	C(12)—O(12)—C(13)	116·6 (3)
C(5)—N(6)—C(7)	116·1 (3)	O(12)—C(13)—O(13)	123·0 (4)
C(5)—N(6)—O(11)	104·0 (3)	O(12)—C(13)—C(14)	111·0 (4)
C(7)—N(6)—O(11)	108·3 (3)	O(13)—C(13)—C(14)	126·1 (4)

C(10)—C(1)—O(2)—C(3)	19·6 (4)	C(5)—N(6)—O(11)—C(10)	34·2 (4)
C(10)—C(1)—C(9)—O(8)	−60·3 (4)	C(7)—N(6)—O(11)—C(10)	−89·8 (4)
O(2)—C(1)—C(10)—C(4)	−18·2 (4)	N(6)—C(7)—O(8)—C(9)	−72·1 (4)
C(9)—C(1)—C(10)—O(11)	−15·4 (5)	C(7)—O(8)—C(9)—C(1)	91·2 (4)
C(1)—O(2)—C(3)—C(4)	−12·9 (5)	C(1)—C(9)—C(12)—O(12)	−42·7 (4)
O(2)—C(3)—C(4)—C(10)	0·5 (5)	C(1)—C(10)—O(11)—N(6)	86·0 (4)
C(10)—C(4)—C(5)—N(6)	9·1 (4)	C(4)—C(10)—O(11)—N(6)	−28·7 (4)
C(3)—C(4)—C(10)—C(1)	10·9 (5)	C(9)—C(12)—O(12)—C(13)	−67·0 (4)
C(5)—C(4)—C(10)—O(11)	11·4 (4)	C(12)—O(12)—C(13)—O(13)	1·7 (5)
C(4)—C(5)—N(6)—O(11)	−26·0 (4)	C(12)—O(12)—C(13)—C(14)	−178·9 (3)
O(11)—N(6)—C(7)—O(8)	64·2 (4)		

atomic coordinates are given in Table 1.* Bond distances, bond angles and torsion angles are given in Table 2. Atom numbering is shown in Fig. 1, unit-cell contents in Fig. 2.

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

* Lists of structure factors, anisotropic thermal parameters for the non-H atoms, positional and isotropic thermal parameters for the H atoms and a table of the shortest non-bonded contacts have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51964 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

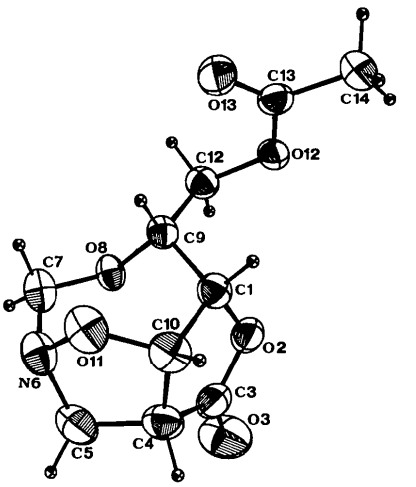

Fig. 1. *ORTEP*II (Johnson, 1976) view of the molecules with the atom numbering. The thermal ellipsoids are given at 50% probability.

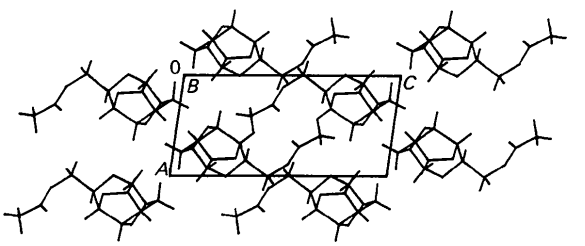

Fig. 2. Packing diagram.

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