

1,2;5,6-Di-O-isopropylidene-3-C-nitro-methyl- α -D-allofuranose

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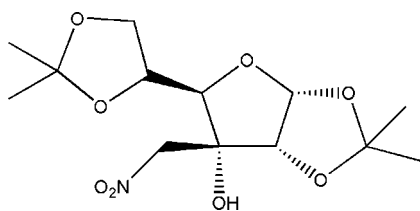
Received 4 May 2011; accepted 7 May 2011

Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.061; wR factor = 0.164; data-to-parameter ratio = 7.9.

The molecule of the title compound, $\text{C}_{13}\text{H}_{21}\text{NO}_8$, consists of two methylenedioxy rings and one tetrahydrofuran ring. In the crystal, intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into helical chains running along the 6_1 screw axis. Weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds help to stabilize the crystal packing. Voids of 245 Å³ per unit cell occur.

Related literature

For details of the synthesis, see: Saito *et al.* (2002). For recent studies of the biological activity of azasugars, see: Loiseleur *et al.* (2007); Rahman *et al.* (2008).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{21}\text{NO}_8$
 $M_r = 319.31$
 Hexagonal, $P6_1$
 $a = 13.2581$ (19) Å

$c = 16.462$ (3) Å
 $V = 2506.0$ (7) Å³
 $Z = 6$
 Mo $K\alpha$ radiation

$\mu = 0.11$ mm⁻¹
 $T = 291$ K

$0.24 \times 0.20 \times 0.20$ mm

Data collection

Rigaku R-AXIS-IV diffractometer
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.975$, $T_{\max} = 0.979$

8380 measured reflections
 1612 independent reflections
 1534 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.047$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.164$
 $S = 1.08$
 1612 reflections
 205 parameters
 1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.43$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O3}-\text{H3E}\cdots\text{O6}^i$	0.90 (8)	1.95 (8)	2.814 (5)	161 (7)
$\text{C1}-\text{H1A}\cdots\text{O3}^{ii}$	0.98	2.37	3.258 (4)	151
$\text{C5}-\text{H5A}\cdots\text{O1}^{iii}$	0.98	2.50	3.320 (4)	141

Symmetry codes: (i) $x - y, x, z + \frac{1}{6}$; (ii) $x - y + 1, x + 1, z + \frac{1}{6}$; (iii) $-x + y, -x + 1, z - \frac{1}{3}$.

Data collection: *R-AXIS-IV Software* (Rigaku, 1997); cell refinement: *R-AXIS-IV Software*; data reduction: *R-AXIS-IV Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *TEXSAN* (Molecular Structure Corporation, 1992); software used to prepare material for publication: *TEXSAN*.

We gratefully acknowledge financial support by the National Natural Science Foundation of China (grant No. 20572103).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5090).

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supplementary materials

Acta Cryst. (2011). E67, o1402 [doi:10.1107/S1600536811017314]

1,2;5,6-Di-*O*-isopropylidene-3-*C*-nitromethyl- α -*D*-allofuranose

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Comment

Asasugars were recently used as novel glycosyls to synthesize novel *N*-nucleosides (Loiseleur *et al.*, 2007; Rahman *et al.*, 2008). Herewith we report the synthesis and crystal structure of the title compound (I) prepared in enantiomerically pure form from 1,2;5,6-di-*O*-isopropylidene-3-carbonyl- α -*D*-glucofuranose (Saito *et al.*, 2002) at room temperature, whose raw material was D-glucose.

The molecule of (I) consists of two methylenedioxy rings and one tetrahydrofuran ring (Fig. 1). In (I), the tetrahydrofuran ring fuses with one methylenedioxy ring, having the *cis* arrangement at the ring junctions and giving a V-shaped molecule. The angles O1—C8—O2, O5—C11—O6, C9—C8—C10 and C12—C11—C13 around the two isopropylidenes are 104.1 (4), 105.5 (4), 113.9 (5) and 114.2 (6)°, respectively.

In the crystal structure, intermolecular O—H \cdots O hydrogen bonds (Table 1) link the molecules into helical chains running along screw axis 6₁, and weak intermolecular C—H \cdots O hydrogen bonds (Table 1) help to stabilize the crystal packing.

Experimental

All reagents and solvents were used as obtained without further purification. 1,2;5,6-di-*O*-isopropylidene-3-*C*-(nitromethyl)- α -*D*-allofuranose was synthesized from 1,2;5,6-di-*O*-isopropylidene-3-carbonyl- α -*D*-glucofuranose as described previously by Saito *et al.* (2002), whose starting material was D-glucose. To a solution of 1,2;5,6-di-*O*-isopropylidene-3-carbonyl- α -*D*-Glucofuranose (7.0 g, 27 mmol) in tetrahydrofuran (50 ml) was added CH₃NO₂ (10.5 ml) and potassium fluoride (3.0 g). The mixture was stirred at room temperature for 6 h. The reaction mixture was then concentrated *in vacuo* and extracted with water and EtOAc, dried (Na₂SO₄), and evaporated. The residue was recrystallized in CH₃OH to yield the title compound. Crystals suitable for X-ray analysis were grown by slow evaporation from methanol at room temperature for two weeks. *R*_f = 0.7 (CHCl₃/EtOAc, 7:3); mp: 110–111°C, [α]_D²⁰ = +96° (c, 1.0, CH₃OH); ¹H NMR (400 MHz, CDCl₃) σ : 5.85 (1H, d, *J* = 3.6 Hz), 4.97 (1H, d, *J* = 12 Hz), 4.89 (1H, d, *J* = 3.6 Hz), 4.49 (1H, d, *J* = 12 Hz), 4.13 (1H, m), 4.01 (1H, m), 3.95 (1H, m), 3.89 (1H, d, *J* = 8.8), 3.27 (1H, s), 1.61 (3H, s), 1.47 (3H, s), 1.39 (3H, s), 1.36 (3H, s); ¹³C NMR (100 MHz, CDCl₃) σ : 113.3, 110.4, 103.7, 81.7, 79.8, 78.5, 77.6, 72.9, 67.9, 26.6, 26.5, 26.5, 25.0.

Refinement

Atom H3E was located on a difference map and isotropically refined. C-bound H atoms were placed geometrically and treated as riding on their parent atoms with C—H are 0.96 Å (methylene) or 0.93 Å (aromatic), and *U*_{iso}(H) = 1.2*U*_{eq}(C). In the absence of any significant anomalous scatterers in the molecule, attempts to confirm the absolute structure by refinement of the Flack parameter in the presence of 1468 sets of Friedel equivalents led to an inconclusive value of 10 (10). Therefore, the Friedel pairs were merged before the final refinement and the absolute configuration was assigned to correspond with that

of the known chiral centres in a precursor molecule, which remained unchanged during the synthesis of the title compound. The porous crystal packing exhibits voids of 245 Å³.

Figures

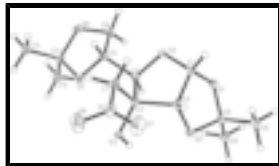


Fig. 1. The molecular structure of (I) showing the atomic numbering and 30% probability displacement ellipsoids. H atoms omitted for clarity.

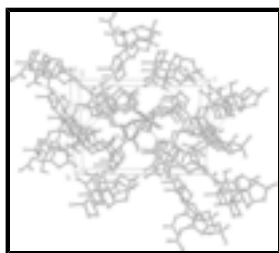


Fig. 2. Packing diagram.

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Crystal data

C₁₃H₂₁NO₈

$M_r = 319.31$

Hexagonal, $P6_1$

Hall symbol: P 61

$a = 13.2581 (19) \text{ \AA}$

$c = 16.462 (3) \text{ \AA}$

$V = 2506.0 (7) \text{ \AA}^3$

$Z = 6$

$F(000) = 1020$

$D_x = 1.270 \text{ Mg m}^{-3}$

Melting point = 383–384 K

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 398 reflections

$\theta = 2\text{--}25.1^\circ$

$\mu = 0.11 \text{ mm}^{-1}$

$T = 291 \text{ K}$

Prismatic, colourless

$0.24 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Rigaku R-Axis-IV
diffractometer

Radiation source: fine-focus sealed tube
graphite

Detector resolution: 0 pixels mm⁻¹

Oscillation frames scans

Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.975$, $T_{\max} = 0.979$

8380 measured reflections

1612 independent reflections

1534 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.047$

$\theta_{\max} = 25.5^\circ$, $\theta_{\min} = 1.8^\circ$

$h = -13 \rightarrow 16$

$k = -16 \rightarrow 0$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.061$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.164$	$w = 1/[\sigma^2(F_o^2) + (0.0962P)^2 + 1.0744P]$
$S = 1.08$	where $P = (F_o^2 + 2F_c^2)/3$
1612 reflections	$(\Delta/\sigma)_{\max} < 0.001$
205 parameters	$\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kF_c[1+0.001xF_c^2\lambda^3/\sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.008 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.2338 (3)	0.7097 (4)	0.2334 (2)	0.0565 (10)
O2	0.0552 (3)	0.6458 (3)	0.1793 (2)	0.0427 (8)
O3	0.0092 (2)	0.5408 (3)	0.0319 (2)	0.0405 (7)
O4	0.3029 (3)	0.6935 (3)	0.1082 (2)	0.0490 (9)
O5	0.1802 (3)	0.4394 (3)	-0.0205 (3)	0.0597 (11)
O6	0.3601 (3)	0.4537 (4)	-0.0197 (3)	0.0557 (9)
O7	0.0437 (7)	0.7865 (7)	-0.0252 (4)	0.127 (3)
O8	0.0234 (7)	0.6912 (7)	-0.1261 (4)	0.131 (3)
N1	0.0764 (5)	0.7349 (5)	-0.0646 (3)	0.0639 (13)
C1	0.2559 (4)	0.7508 (4)	0.1523 (3)	0.0472 (12)
H1A	0.3068	0.8355	0.1502	0.057*
C2	0.1347 (4)	0.7135 (4)	0.1170 (3)	0.0417 (10)
H2A	0.1263	0.7800	0.1005	0.050*
C3	0.1227 (4)	0.6337 (4)	0.0451 (3)	0.0358 (9)
C4	0.2095 (3)	0.5932 (4)	0.0705 (3)	0.0362 (10)

supplementary materials

H4A	0.1721	0.5317	0.1114	0.043*
C5	0.2641 (4)	0.5535 (5)	0.0066 (3)	0.0445 (11)
H5A	0.2918	0.6081	-0.0392	0.053*
C6	0.3605 (5)	0.5349 (5)	0.0379 (3)	0.0514 (12)
H6A	0.4347	0.6070	0.0381	0.062*
H6B	0.3438	0.5025	0.0923	0.062*
C7	0.1610 (4)	0.7012 (5)	-0.0346 (3)	0.0496 (12)
H7A	0.2360	0.7709	-0.0268	0.059*
H7B	0.1703	0.6537	-0.0754	0.059*
C8	0.1192 (4)	0.6842 (5)	0.2544 (3)	0.0463 (12)
C9	0.0671 (6)	0.5833 (6)	0.3133 (4)	0.0650 (15)
H9A	0.0672	0.5174	0.2892	0.097*
H9B	-0.0114	0.5635	0.3258	0.097*
H9C	0.1125	0.6050	0.3623	0.097*
C10	0.1218 (6)	0.7930 (6)	0.2860 (4)	0.0693 (17)
H10A	0.1542	0.8528	0.2453	0.104*
H10B	0.1689	0.8196	0.3341	0.104*
H10C	0.0440	0.7755	0.2986	0.104*
C11	0.2428 (4)	0.3839 (5)	-0.0501 (4)	0.0552 (13)
C12	0.1872 (7)	0.2647 (6)	-0.0156 (6)	0.093 (2)
H12A	0.1886	0.2690	0.0426	0.139*
H12B	0.2291	0.2269	-0.0332	0.139*
H12C	0.1080	0.2210	-0.0340	0.139*
C13	0.2482 (6)	0.3881 (7)	-0.1415 (4)	0.0729 (19)
H13A	0.2860	0.4677	-0.1592	0.109*
H13B	0.1707	0.3469	-0.1632	0.109*
H13C	0.2915	0.3523	-0.1602	0.109*
H3E	-0.017 (6)	0.497 (6)	0.077 (5)	0.067 (19)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0408 (19)	0.077 (3)	0.0453 (19)	0.0249 (17)	-0.0092 (15)	-0.0111 (18)
O2	0.0312 (16)	0.0491 (19)	0.0426 (17)	0.0161 (15)	-0.0026 (13)	-0.0029 (14)
O3	0.0244 (15)	0.0419 (17)	0.0460 (17)	0.0098 (14)	-0.0045 (13)	0.0004 (15)
O4	0.0269 (16)	0.057 (2)	0.054 (2)	0.0135 (14)	-0.0040 (14)	-0.0109 (16)
O5	0.0323 (17)	0.066 (2)	0.075 (2)	0.0197 (16)	-0.0017 (17)	-0.028 (2)
O6	0.045 (2)	0.071 (2)	0.059 (2)	0.0357 (19)	0.0046 (17)	-0.0059 (19)
O7	0.193 (8)	0.185 (7)	0.089 (4)	0.159 (7)	-0.029 (4)	-0.013 (5)
O8	0.173 (6)	0.184 (7)	0.094 (4)	0.132 (6)	-0.057 (5)	-0.031 (5)
N1	0.095 (4)	0.069 (3)	0.044 (3)	0.053 (3)	-0.006 (3)	0.003 (2)
C1	0.033 (2)	0.041 (3)	0.056 (3)	0.010 (2)	-0.005 (2)	-0.010 (2)
C2	0.037 (2)	0.037 (2)	0.047 (3)	0.016 (2)	-0.003 (2)	0.002 (2)
C3	0.029 (2)	0.034 (2)	0.040 (2)	0.0126 (18)	-0.0018 (17)	0.0001 (18)
C4	0.026 (2)	0.040 (2)	0.035 (2)	0.0113 (18)	-0.0010 (17)	0.0023 (18)
C5	0.034 (2)	0.054 (3)	0.043 (2)	0.020 (2)	0.0006 (19)	-0.002 (2)
C6	0.044 (3)	0.067 (3)	0.047 (3)	0.031 (3)	0.000 (2)	-0.008 (3)
C7	0.041 (3)	0.058 (3)	0.046 (3)	0.022 (2)	0.003 (2)	0.011 (2)

C8	0.035 (2)	0.055 (3)	0.043 (3)	0.018 (2)	-0.0061 (19)	-0.011 (2)
C9	0.063 (4)	0.077 (4)	0.050 (3)	0.031 (3)	-0.006 (3)	0.003 (3)
C10	0.067 (4)	0.070 (4)	0.067 (4)	0.031 (3)	0.004 (3)	-0.019 (3)
C11	0.044 (3)	0.064 (3)	0.059 (3)	0.027 (3)	0.012 (2)	-0.010 (3)
C12	0.093 (5)	0.065 (4)	0.106 (6)	0.028 (4)	0.033 (5)	0.000 (4)
C13	0.060 (3)	0.105 (5)	0.061 (3)	0.047 (4)	-0.005 (3)	-0.023 (4)

Geometric parameters (Å, °)

O1—C1	1.415 (7)	C5—C6	1.509 (7)
O1—C8	1.424 (6)	C5—H5A	0.9800
O2—C2	1.421 (6)	C6—H6A	0.9700
O2—C8	1.441 (6)	C6—H6B	0.9700
O3—C3	1.405 (5)	C7—H7A	0.9700
O3—H3E	0.90 (8)	C7—H7B	0.9700
O4—C1	1.402 (6)	C8—C9	1.510 (8)
O4—C4	1.428 (5)	C8—C10	1.517 (8)
O5—C5	1.429 (6)	C9—H9A	0.9600
O5—C11	1.441 (6)	C9—H9B	0.9600
O6—C6	1.432 (6)	C9—H9C	0.9600
O6—C11	1.445 (7)	C10—H10A	0.9600
O7—N1	1.171 (8)	C10—H10B	0.9600
O8—N1	1.204 (8)	C10—H10C	0.9600
N1—C7	1.484 (7)	C11—C12	1.483 (10)
C1—C2	1.540 (7)	C11—C13	1.506 (9)
C1—H1A	0.9800	C12—H12A	0.9600
C2—C3	1.542 (7)	C12—H12B	0.9600
C2—H2A	0.9800	C12—H12C	0.9600
C3—C7	1.525 (7)	C13—H13A	0.9600
C3—C4	1.551 (6)	C13—H13B	0.9600
C4—C5	1.513 (6)	C13—H13C	0.9600
C4—H4A	0.9800		
C1—O1—C8	108.2 (4)	H6A—C6—H6B	109.2
C2—O2—C8	106.1 (3)	N1—C7—C3	112.5 (4)
C3—O3—H3E	110 (4)	N1—C7—H7A	109.1
C1—O4—C4	108.4 (3)	C3—C7—H7A	109.1
C5—O5—C11	107.7 (4)	N1—C7—H7B	109.1
C6—O6—C11	108.0 (4)	C3—C7—H7B	109.1
O7—N1—O8	116.7 (7)	H7A—C7—H7B	107.8
O7—N1—C7	123.3 (5)	O1—C8—O2	104.1 (4)
O8—N1—C7	118.8 (6)	O1—C8—C9	109.1 (4)
O4—C1—O1	110.1 (4)	O2—C8—C9	108.1 (4)
O4—C1—C2	107.8 (4)	O1—C8—C10	110.0 (4)
O1—C1—C2	104.5 (4)	O2—C8—C10	111.1 (5)
O4—C1—H1A	111.4	C9—C8—C10	113.9 (5)
O1—C1—H1A	111.4	C8—C9—H9A	109.5
C2—C1—H1A	111.4	C8—C9—H9B	109.5
O2—C2—C1	104.8 (4)	H9A—C9—H9B	109.5
O2—C2—C3	109.3 (4)	C8—C9—H9C	109.5

supplementary materials

C1—C2—C3	104.0 (4)	H9A—C9—H9C	109.5
O2—C2—H2A	112.7	H9B—C9—H9C	109.5
C1—C2—H2A	112.7	C8—C10—H10A	109.5
C3—C2—H2A	112.7	C8—C10—H10B	109.5
O3—C3—C7	106.1 (4)	H10A—C10—H10B	109.5
O3—C3—C2	114.6 (4)	C8—C10—H10C	109.5
C7—C3—C2	111.6 (4)	H10A—C10—H10C	109.5
O3—C3—C4	113.2 (3)	H10B—C10—H10C	109.5
C7—C3—C4	110.4 (4)	O5—C11—O6	105.5 (4)
C2—C3—C4	101.0 (3)	O5—C11—C12	108.0 (5)
O4—C4—C5	106.5 (3)	O6—C11—C12	110.6 (6)
O4—C4—C3	104.1 (3)	O5—C11—C13	110.4 (6)
C5—C4—C3	119.9 (4)	O6—C11—C13	107.8 (5)
O4—C4—H4A	108.6	C12—C11—C13	114.2 (6)
C5—C4—H4A	108.6	C11—C12—H12A	109.5
C3—C4—H4A	108.6	C11—C12—H12B	109.5
O5—C5—C6	102.0 (4)	H12A—C12—H12B	109.5
O5—C5—C4	109.4 (4)	C11—C12—H12C	109.5
C6—C5—C4	114.2 (4)	H12A—C12—H12C	109.5
O5—C5—H5A	110.3	H12B—C12—H12C	109.5
C6—C5—H5A	110.3	C11—C13—H13A	109.5
C4—C5—H5A	110.3	C11—C13—H13B	109.5
O6—C6—C5	102.2 (4)	H13A—C13—H13B	109.5
O6—C6—H6A	111.3	C11—C13—H13C	109.5
C5—C6—H6A	111.3	H13A—C13—H13C	109.5
O6—C6—H6B	111.3	H13B—C13—H13C	109.5
C5—C6—H6B	111.3		
C4—O4—C1—O1	91.7 (4)	C11—O5—C5—C4	-154.2 (4)
C4—O4—C1—C2	-21.7 (5)	O4—C4—C5—O5	166.8 (4)
C8—O1—C1—O4	-132.3 (4)	C3—C4—C5—O5	-75.7 (5)
C8—O1—C1—C2	-16.8 (5)	O4—C4—C5—C6	53.2 (5)
C8—O2—C2—C1	24.8 (5)	C3—C4—C5—C6	170.8 (4)
C8—O2—C2—C3	135.8 (4)	C11—O6—C6—C5	-29.7 (6)
O4—C1—C2—O2	111.9 (4)	O5—C5—C6—O6	37.9 (5)
O1—C1—C2—O2	-5.2 (5)	C4—C5—C6—O6	155.7 (4)
O4—C1—C2—C3	-2.9 (5)	O7—N1—C7—C3	53.9 (9)
O1—C1—C2—C3	-119.9 (4)	O8—N1—C7—C3	-112.8 (7)
O2—C2—C3—O3	34.0 (5)	O3—C3—C7—N1	53.1 (6)
C1—C2—C3—O3	145.5 (4)	C2—C3—C7—N1	-72.5 (5)
O2—C2—C3—C7	154.7 (4)	C4—C3—C7—N1	176.1 (4)
C1—C2—C3—C7	-93.8 (5)	C1—O1—C8—O2	32.4 (5)
O2—C2—C3—C4	-88.0 (4)	C1—O1—C8—C9	147.7 (4)
C1—C2—C3—C4	23.5 (4)	C1—O1—C8—C10	-86.7 (5)
C1—O4—C4—C5	164.9 (4)	C2—O2—C8—O1	-35.5 (5)
C1—O4—C4—C3	37.3 (5)	C2—O2—C8—C9	-151.4 (4)
O3—C3—C4—O4	-159.9 (4)	C2—O2—C8—C10	82.9 (5)
C7—C3—C4—O4	81.3 (4)	C5—O5—C11—O6	15.2 (6)
C2—C3—C4—O4	-36.8 (4)	C5—O5—C11—C12	133.6 (6)
O3—C3—C4—C5	81.4 (5)	C5—O5—C11—C13	-100.9 (5)

C7—C3—C4—C5	-37.4 (5)	C6—O6—C11—O5	10.1 (6)
C2—C3—C4—C5	-155.6 (4)	C6—O6—C11—C12	-106.5 (6)
C11—O5—C5—C6	-32.9 (5)	C6—O6—C11—C13	128.0 (5)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O3—H3E \cdots O6 ⁱ	0.90 (8)	1.95 (8)	2.814 (5)	161 (7)
C1—H1A \cdots O3 ⁱⁱ	0.98	2.37	3.258 (4)	151.
C5—H5A \cdots O1 ⁱⁱⁱ	0.98	2.50	3.320 (4)	141.

Symmetry codes: (i) $x-y, x, z+1/6$; (ii) $x-y+1, x+1, z+1/6$; (iii) $-x+y, -x+1, z-1/3$.

Fig. 1

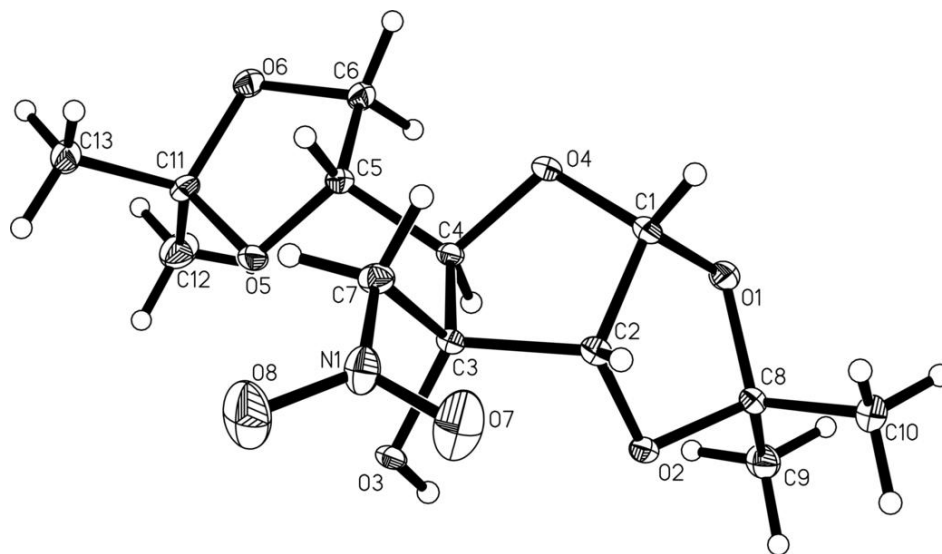


Fig. 2

