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Crystal structure of 3-deoxy-3-nitromethyl-1,2;5,6-di-*O*-isopropylidene- α -D-allofuranose

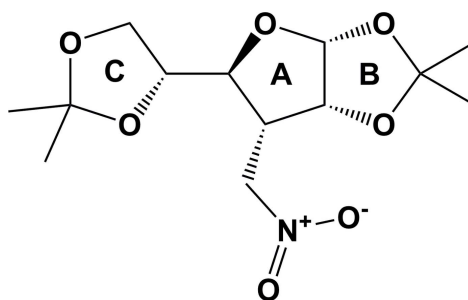
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The title compound, C₁₃H₂₁NO₇ [systematic name: (3*aR*,5*S*,6*R*,6*aR*)-5-[(*R*)-2,2-dimethyl-1,3-dioxolan-4-yl]-2,2-dimethyl-6-(nitromethyl)tetrahydrofuro[2,3-*d*]-[1,3]dioxole], consists of a substituted 2,2-dimethyltetrahydrofuro[2,3-*d*][1,3]-dioxolane skeleton. The furanose ring *A* adopts a ^oT₄ conformation. The fused dioxolane ring *B* and the substituent dioxolane ring *C* also have twisted conformations. There are no strong hydrogen bonds in the crystal structure: only weak C—H...O contacts are present, which link the molecules to form a three-dimensional structure.

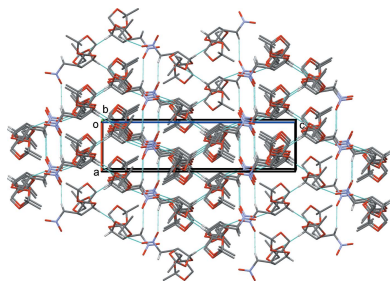
1. Chemical context

The title compound **1**, has been used for the syntheses of isoxazoles (Luginina *et al.*, 2013) and carbohydrate-based amines and amino acids (Rjabovs *et al.*, 2015*a*). Carbohydrates with amino groups are valuable synthetic precursors and are easily converted to spirocyclic carbohydrate derivatives (Turks *et al.*, 2013), imino sugars (Filichev & Pedersen, 2001), nucleic acid mimetics (Rozners *et al.*, 2003), and azido sugars (Mackeviča *et al.*, 2014; Rjabova *et al.*, 2012). The latter are widely used for the syntheses of triazoles (Uzuleņa *et al.*, 2015; Grigorjeva *et al.*, 2015) and THF-amino acids (sugar amino acids) (Rjabovs & Turks, 2013).



2. Structural commentary

The title compound **1**, consists of a tetrahydrofuran core (ring *A*) fused with a dioxolane ring (*B*) and substituted with a dioxolane (ring *C*) and a nitromethyl group (Fig. 1). The conformational analysis of the furanose ring (*A*) based on the internal dihedral angles of the ring shows that its pseudo-rotational phase angle $P = 70^\circ$ (Altona & Sundaralingam, 1972; Taha *et al.*, 2013). Thus, this ring adopts a conformation close to ^oT₄, where O1 and C4 deviate by 0.214 (2) and −0.340 (3) Å, respectively, from the plane through atoms



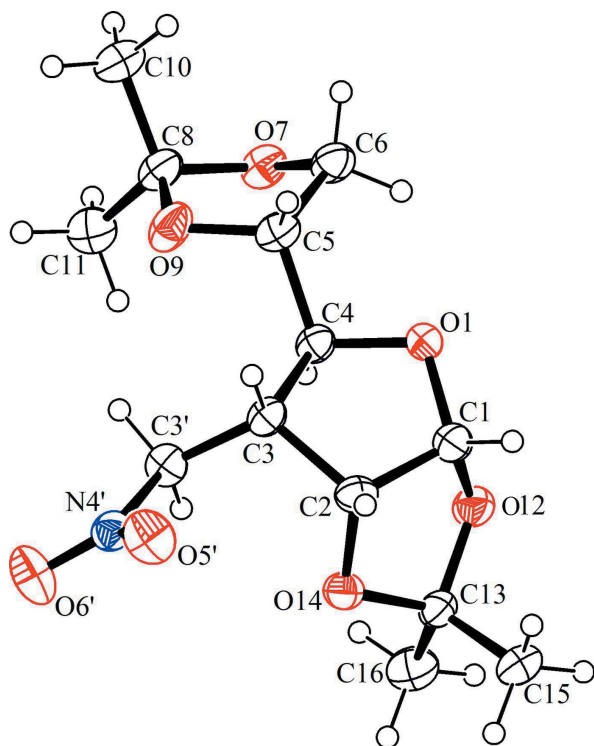


Figure 1
The molecular structure of the title compound **1**, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

C1/C2/C3. Such a conformation of the furanose ring is rather unusual for 3-*C*-monosubstituted 3-deoxy-1,2-*O*-isopropylidene- α -*D*-allofuranoses. For example, previously reported structures (Rjabovs *et al.*, 2014, 2015*a,b*) had conformations between 3E and 3T_4 . The fused dioxolane ring *B* also adopts a twisted conformation on bond C13—O12; these atoms deviate by -0.324 (4) and 0.224 (3) Å, respectively, from plane C1/C2/O14. The dihedral angle subtended by the mean planes of rings *A* and *B* is 63.7 (2)°. The five-membered ring of the 2,2-

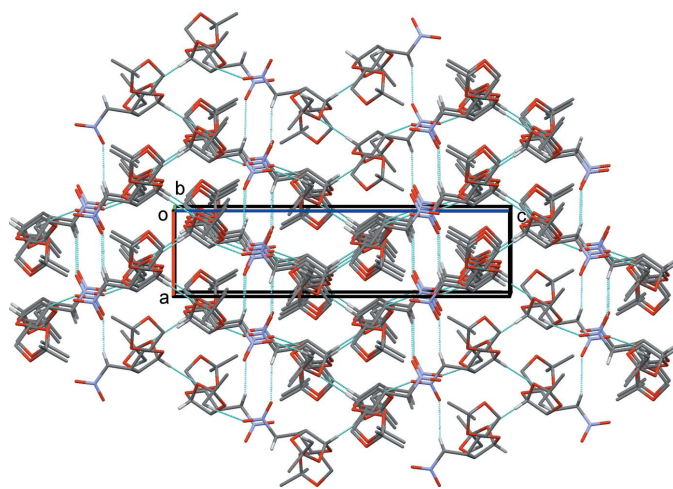


Figure 2
A view along the *b* axis of the crystal packing of the title compound **1**. Hydrogen bonds are shown as dashed lines (see Table 1) and H atoms not involved in these interactions have been omitted for clarity.

Table 1
Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C3'—H3'2···O5' ⁱ	0.97	2.53	3.355 (4)	143
C1—H1···O12 ⁱⁱ	0.98	2.41	3.386 (5)	178
C15—H15A···O6 ⁱⁱⁱ	0.96	2.48	3.433 (5)	174

Symmetry codes: (i) $x - 1, y, z$; (ii) $x + \frac{1}{2}, -y + \frac{3}{2}, -z$; (iii) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$

dimethyl-1,3-dioxolan-4-yl group, ring *C*, also adopts a twisted conformation, on bond C6—O7; these atoms deviate by 0.143 (4) and -0.381 (2) Å, respectively from plane C5/O9/C8.

3. Supramolecular features

In the crystal, molecules are linked *via* C—H···O hydrogen bonds, forming chains along [100]. The chains are linked *via* further C—H···O hydrogen bonds, forming a three-dimensional structure (Table 1 and Fig. 2).

4. Database survey

A search of the Cambridge Structural Database (Version 5.37; Groom & Allen, 2014) for substructure S1 (Fig. 3) gave 137 hits, while a search for substructure S2 (Fig. 3) gave only five hits. Amongst the latter compounds, four concern the structures with a hydroxyl and a nitromethyl group attached to atom C3 (BOGFOU: Turks *et al.*, 2014; BOGFUA: Turks *et al.*, 2014; CIDVO: Turks *et al.*, 2013; USODEM: Zhang *et al.*, 2011). They are diastereomers crystallizing in space groups $P2_12_12_1$, $P3_2$, $C2$ and $P6_1$, respectively. In the fifth compound (KATWIN; Lugiņina *et al.*, 2012), the extra substituent at atom C3 is a methylthio group; it crystallizes in space group $C2$.

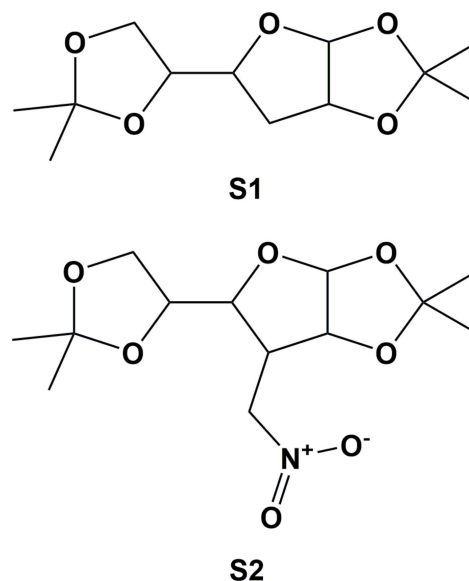


Figure 3
Substructures used for the database survey.

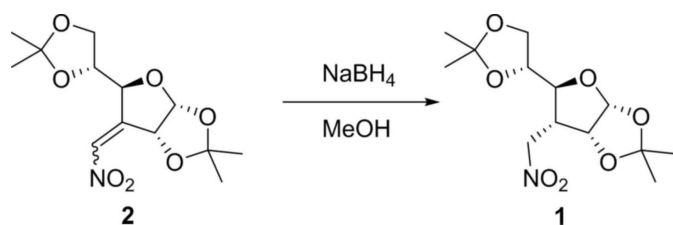


Figure 4
Synthesis of the title compound.

5. Synthesis and crystallization

The title compound **1**, was synthesized by reduction of the nitro olefin **2** (Albrecht & Moffatt, 1970; Filichev *et al.*, 2001; Lugiņina *et al.*, 2012) with sodium borohydride in methanol solution, as illustrated in Fig. 4. NaBH₄ (6.2 g, 163.9 mmol, 5.5 eq.) was added portion wise to a solution of **2** (9.1 g, 30.0 mmol, 1.0 eq.) in MeOH (200 ml) over 30 min at 273 K. After completion (monitored by TLC) the reaction mixture was acidified using 10% aqueous solution of AcOH to pH 6–7 and then evaporated to dryness. The residue was dissolved in EtOAc (90 ml), washed with brine (3 × 10 ml), dried over NaSO₄, and evaporated. The product **1** was purified by column chromatography on silica gel (Hexanes/EtOAc 3:1 → 2:1) giving a white crystalline solid (yield: 6.5 g, 72%; m.p. 355–356 K). *R_f* = 0.9 (hexanes/EtOAc 1:1). ¹H NMR (CDCl₃, 300 MHz): δ 5.84 (*d*, *J* = 3.7 Hz, 1H), 4.88–4.82 (*m*, 2H), 4.68 (*dd*, AB syst., *J* = 14.9 Hz, *J* = 10.4 Hz, 1H), 4.14 (*dd*, *J* = 8.0 Hz, *J* = 5.5 Hz, 1H), 4.02–3.92 (*m*, 2H), 3.65 (*dd*, *J* = 9.9 Hz, *J* = 8.4 Hz, 1H), 2.74 (*tt*, *J* = 10.1 Hz, *J* = 4.4 Hz, 1H), 1.52 (*s*, 3H), 1.40 (*s*, 3H), 1.33 (*s*, 3H), 1.32 (*s*, 3H). ¹³C-NMR (75 MHz, CDCl₃): δ 112.6, 110.1, 105.4, 80.5, 79.0, 77.8, 70.8, 68.2, 46.6, 26.8, 26.7, 26.4, 25.1. X-ray quality single crystals were obtained by slow evaporation of a dichloromethane solution at ambient temperature.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The H atoms were included in calculated positions and refined as riding atoms: C–H = 0.96–0.98 Å with *U*_{iso}(H) = 1.5*U*_{eq}(C-methyl) and 1.2*U*_{eq}(C) for other H atoms. The absolute configuration is based on that of the starting material.

Acknowledgements

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References

- Albrecht, H. P. & Moffatt, J. G. (1970). *Tetrahedron Lett.* **11**, 1063–1066.
 Altona, C. & Sundaralingam, M. (1972). *J. Am. Chem. Soc.* **94**, 8205–8212.

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₃ H ₂₁ NO ₇
<i>M_r</i>	303.31
Crystal system, space group	Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁
Temperature (K)	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	5.5044 (2), 12.6144 (4), 21.6348 (9)
<i>V</i> (Å ³)	1502.21 (10)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
<i>μ</i> (mm ⁻¹)	0.11
Crystal size (mm)	0.26 × 0.08 × 0.06
Data collection	
Diffractionmeter	Nonius KappaCCD
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	4225, 4225, 2316
(sin θ/λ) _{max} (Å ⁻¹)	0.705
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.065, 0.127, 1.01
No. of reflections	4225
No. of parameters	194
H-atom treatment	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.20, −0.26

Computer programs: *KappaCCD Server Software* (Nonius, 1997), *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997), *SIR2011* (Burla *et al.*, 2012), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

- Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., Giacovazzo, C., Mallamo, M., Mazzone, A., Polidori, G. & Spagna, R. (2012). *J. Appl. Cryst.* **45**, 357–361.
 Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
 Filichev, V. V., Brandt, M. & Pedersen, E. B. (2001). *Carbohydr. Res.* **333**, 115–122.
 Filichev, V. V. & Pedersen, E. B. (2001). *Tetrahedron*, **57**, 9163–9168.
 Grigorjeva, J., Uzuleņa, J., Rjabovs, V. & Turks, M. (2015). *Chem. Heterocycl. Compd.* **51**, 883–890.
 Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.
 Lugiņina, J., Rjabovs, V., Belyakov, S. & Turks, M. (2012). *Carbohydr. Res.* **350**, 86–89.
 Lugiņina, J., Rjabovs, V., Belyakov, S. & Turks, M. (2013). *Tetrahedron Lett.* **54**, 5328–5331.
 Mackeviča, J., Ostrovskis, P., Leffler, H., Nilsson, U. J., Rudovica, V., Viksna, A., Belyakov, S. & Turks, M. (2014). *ARKIVOC*, (iii), 90–112.
 Nonius (1997). *KappaCCD Server Software*. Nonius BV, Delft, The Netherlands.
 Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
 Rjabova, J., Rjabovs, V., Moreno Vargas, A. J., Clavijo, E. M. & Turks, M. (2012). *Cent. Eur. J. Chem.* **10**, 386–394.
 Rjabovs, V., Mishnev, A., Kiselovs, G. & Turks, M. (2014). *Acta Cryst. E* **70**, o524–o525.
 Rjabovs, V., Ostrovskis, P., Posevins, D., Kiselovs, G., Kumpiņš, V., Mishnev, A. & Turks, M. (2015a). *Eur. J. Org. Chem.* pp. 5572–5584.
 Rjabovs, V., Stepanovs, D. & Turks, M. (2015b). *Acta Cryst. E* **71**, 1212–1215.
 Rjabovs, V. & Turks, M. (2013). *Tetrahedron*, **69**, 10693–10710.
 Rozners, E., Katkevica, D., Bizdena, E. & Strömberg, R. (2003). *J. Am. Chem. Soc.* **125**, 12125–12136.
 Sheldrick, G. M. (2015). *Acta Cryst. C* **71**, 3–8.
 Spek, A. L. (2009). *Acta Cryst. D* **65**, 148–155.
 Taha, H. A., Richards, M. R. & Lowary, T. L. (2013). *Chem. Rev.* **113**, 1851–1876.

- Turks, M., Rodins, V., Rolava, E., Ostrovskis, P. & Belyakov, S. (2013). *Carbohydr. Res.* **375**, 5–15.
- Turks, M., Vēze, K., Kiselovs, G., Mackeviča, J., Lugiņina, J., Mishnev, A. & Markovič, D. (2014). *Carbohydr. Res.* **391**, 82–88.
- Uzuleņa, J., Rjabovs, V., Moreno-Vargas, A. J. & Turks, M. (2015). *Chem. Heterocycl. Compd.* **51**, 664–671.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Zhang, Q., Ke, Y., Cheng, W., Li, P. & Liu, H. (2011). *Acta Cryst.* **E67**, o1402.

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Acta Cryst. (2016). E72, 314-317 [doi:10.1107/S2056989016001845]

Crystal structure of 3-deoxy-3-nitromethyl-1,2;5,6-di-*O*-isopropylidene- α -D-allofuranose

Jevgenija Lugiņina, Vitālijs Rjabovs and Dmitrijs Stepanovs

Computing details

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR2011* (Burla *et al.*, 2012); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *pubCIF* (Westrip, 2010).

(3*aR*,5*S*,6*R*,6*aR*)-5-[(*R*)-2,2-Dimethyl-1,3-\ dioxolan-4-yl]-2,2-dimethyl-6-(nitromethyl)tetrahydrofuro[2,3-*d*][1,3]\ dioxole

Crystal data

C₁₃H₂₁NO₇

M_r = 303.31

Orthorhombic, *P*2₁2₁2₁

a = 5.5044 (2) Å

b = 12.6144 (4) Å

c = 21.6348 (9) Å

V = 1502.21 (10) Å³

Z = 4

F(000) = 648

D_x = 1.341 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 26214 reflections

θ = 1.0–30.0°

μ = 0.11 mm⁻¹

T = 173 K

Needle, colourless

0.26 × 0.08 × 0.06 mm

Data collection

Nonius KappaCCD

diffractometer

Radiation source: fine-focus sealed tube

φ and ω scan

4225 measured reflections

4225 independent reflections

2316 reflections with *I* > 2σ(*I*)

θ_{max} = 30.1°, θ_{min} = 3.2°

h = -7→7

k = -17→17

l = -30→30

Refinement

Refinement on *F*²

Least-squares matrix: full

R [*F*² > 2σ(*F*²)] = 0.065

wR(*F*²) = 0.127

S = 1.01

4225 reflections

194 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F*_o²) + (0.0367*P*)² + 0.3523*P*]

where *P* = (*F*_o² + 2*F*_c²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.20 e Å⁻³

Δρ_{min} = -0.25 e Å⁻³

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.1756 (4)	0.94348 (18)	0.03395 (11)	0.0372 (6)
C1	0.2580 (7)	0.8418 (3)	0.04912 (17)	0.0368 (9)
H1	0.3478	0.8095	0.0148	0.044*
C2	0.4157 (6)	0.8508 (3)	0.10747 (17)	0.0339 (8)
H2	0.5873	0.8361	0.0990	0.041*
C3	0.3744 (6)	0.9665 (2)	0.12881 (17)	0.0306 (8)
H3	0.5155	1.0090	0.1166	0.037*
C4	0.1536 (6)	1.0022 (2)	0.09017 (16)	0.0309 (8)
H4	0.0025	0.9832	0.1114	0.037*
C5	0.1502 (6)	1.1182 (3)	0.07239 (18)	0.0360 (9)
H5	0.3020	1.1371	0.0514	0.043*
C6	-0.0661 (6)	1.1468 (3)	0.03179 (19)	0.0392 (9)
H6A	-0.0244	1.2023	0.0026	0.047*
H6B	-0.1248	1.0854	0.0093	0.047*
O7	-0.2414 (4)	1.18276 (17)	0.07540 (12)	0.0391 (6)
C8	-0.1090 (6)	1.2369 (3)	0.12245 (19)	0.0393 (9)
O9	0.1197 (4)	1.18174 (19)	0.12615 (13)	0.0470 (7)
C10	-0.0637 (8)	1.3514 (3)	0.1055 (2)	0.0489 (11)
H10A	-0.2159	1.3882	0.1027	0.073*
H10B	0.0184	1.3548	0.0664	0.073*
H10C	0.0354	1.3840	0.1367	0.073*
C11	-0.2428 (8)	1.2243 (3)	0.18262 (19)	0.0554 (11)
H11A	-0.2530	1.1505	0.1930	0.083*
H11B	-0.4035	1.2530	0.1786	0.083*
H11C	-0.1572	1.2614	0.2147	0.083*
O12	0.0607 (5)	0.77711 (19)	0.06819 (12)	0.0397 (6)
C13	0.1517 (6)	0.7096 (3)	0.11560 (17)	0.0346 (9)
O14	0.3131 (4)	0.77660 (18)	0.14980 (11)	0.0362 (6)
C15	0.2887 (8)	0.6163 (3)	0.08712 (19)	0.0489 (11)
H15A	0.3527	0.5723	0.1194	0.073*
H15B	0.4198	0.6425	0.0621	0.073*
H15C	0.1798	0.5756	0.0619	0.073*
C16	-0.0501 (7)	0.6743 (3)	0.1567 (2)	0.0484 (11)
H16A	-0.1272	0.7352	0.1748	0.073*
H16B	0.0138	0.6299	0.1889	0.073*
H16C	-0.1670	0.6352	0.1330	0.073*
C3'	0.3314 (6)	0.9814 (3)	0.19700 (17)	0.0339 (8)
H3'1	0.2859	1.0546	0.2044	0.041*
H3'2	0.1959	0.9372	0.2095	0.041*

N4'	0.5468 (6)	0.9550 (2)	0.23638 (16)	0.0373 (7)
O5'	0.7368 (4)	0.9282 (2)	0.21135 (13)	0.0518 (8)
O6'	0.5199 (5)	0.9616 (3)	0.29175 (14)	0.0612 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0535 (15)	0.0301 (13)	0.0281 (14)	0.0051 (11)	-0.0038 (12)	-0.0004 (12)
C1	0.045 (2)	0.0324 (19)	0.033 (2)	0.0065 (17)	0.0013 (17)	-0.0011 (16)
C2	0.0328 (19)	0.0309 (18)	0.038 (2)	0.0052 (15)	0.0025 (17)	0.0038 (17)
C3	0.0258 (17)	0.0283 (19)	0.038 (2)	-0.0020 (13)	-0.0002 (15)	-0.0018 (16)
C4	0.0273 (17)	0.0285 (18)	0.037 (2)	-0.0028 (14)	-0.0016 (16)	-0.0007 (17)
C5	0.0299 (18)	0.0302 (19)	0.048 (2)	0.0000 (14)	-0.0025 (18)	0.0015 (18)
C6	0.038 (2)	0.0311 (19)	0.048 (2)	0.0058 (16)	-0.0062 (18)	0.0003 (19)
O7	0.0308 (13)	0.0349 (13)	0.0517 (17)	-0.0016 (11)	-0.0035 (12)	-0.0014 (13)
C8	0.038 (2)	0.0284 (19)	0.052 (3)	0.0024 (15)	-0.0039 (18)	-0.0024 (18)
O9	0.0418 (15)	0.0345 (14)	0.0646 (19)	0.0089 (11)	-0.0173 (13)	-0.0132 (14)
C10	0.055 (2)	0.030 (2)	0.062 (3)	-0.0002 (18)	0.003 (2)	0.003 (2)
C11	0.068 (3)	0.046 (2)	0.052 (3)	-0.004 (2)	0.002 (2)	0.003 (2)
O12	0.0432 (14)	0.0323 (13)	0.0436 (15)	-0.0020 (11)	-0.0114 (12)	0.0004 (13)
C13	0.040 (2)	0.0257 (18)	0.038 (2)	-0.0024 (15)	-0.0072 (17)	-0.0009 (16)
O14	0.0408 (14)	0.0327 (13)	0.0352 (15)	-0.0045 (11)	-0.0058 (12)	0.0045 (12)
C15	0.065 (3)	0.0281 (19)	0.053 (3)	0.0049 (18)	-0.004 (2)	-0.0008 (19)
C16	0.045 (2)	0.042 (2)	0.059 (3)	-0.0028 (18)	0.000 (2)	0.006 (2)
C3'	0.0245 (17)	0.038 (2)	0.040 (2)	-0.0017 (15)	-0.0016 (16)	-0.0010 (17)
N4'	0.0387 (18)	0.0316 (17)	0.042 (2)	-0.0026 (14)	-0.0078 (16)	0.0001 (16)
O5'	0.0312 (14)	0.0693 (19)	0.0548 (19)	0.0061 (14)	-0.0057 (14)	0.0012 (15)
O6'	0.0627 (19)	0.085 (2)	0.0355 (18)	0.0003 (16)	-0.0099 (16)	-0.0045 (17)

Geometric parameters (Å, °)

O1—C1	1.399 (4)	C8—C10	1.511 (5)
O1—C4	1.429 (4)	C10—H10A	0.9600
C1—O12	1.420 (4)	C10—H10B	0.9600
C1—C2	1.537 (5)	C10—H10C	0.9600
C1—H1	0.9800	C11—H11A	0.9600
C2—O14	1.427 (4)	C11—H11B	0.9600
C2—C3	1.547 (5)	C11—H11C	0.9600
C2—H2	0.9800	O12—C13	1.424 (4)
C3—C3'	1.506 (5)	C13—O14	1.432 (4)
C3—C4	1.542 (5)	C13—C16	1.491 (5)
C3—H3	0.9800	C13—C15	1.527 (5)
C4—C5	1.514 (4)	C15—H15A	0.9600
C4—H4	0.9800	C15—H15B	0.9600
C5—O9	1.422 (4)	C15—H15C	0.9600
C5—C6	1.523 (5)	C16—H16A	0.9600
C5—H5	0.9800	C16—H16B	0.9600
C6—O7	1.424 (4)	C16—H16C	0.9600

C6—H6A	0.9700	C3'—N4'	1.498 (4)
C6—H6B	0.9700	C3'—H3'1	0.9700
O7—C8	1.426 (4)	C3'—H3'2	0.9700
C8—O9	1.441 (4)	N4'—O6'	1.210 (4)
C8—C11	1.504 (6)	N4'—O5'	1.225 (4)
C1—O1—C4	107.6 (2)	C11—C8—C10	113.0 (3)
O1—C1—O12	110.3 (3)	C5—O9—C8	109.2 (3)
O1—C1—C2	107.9 (3)	C8—C10—H10A	109.5
O12—C1—C2	103.7 (3)	C8—C10—H10B	109.5
O1—C1—H1	111.5	H10A—C10—H10B	109.5
O12—C1—H1	111.5	C8—C10—H10C	109.5
C2—C1—H1	111.5	H10A—C10—H10C	109.5
O14—C2—C1	104.8 (3)	H10B—C10—H10C	109.5
O14—C2—C3	111.7 (3)	C8—C11—H11A	109.5
C1—C2—C3	103.4 (3)	C8—C11—H11B	109.5
O14—C2—H2	112.1	H11A—C11—H11B	109.5
C1—C2—H2	112.1	C8—C11—H11C	109.5
C3—C2—H2	112.1	H11A—C11—H11C	109.5
C3'—C3—C4	111.8 (3)	H11B—C11—H11C	109.5
C3'—C3—C2	115.7 (3)	C1—O12—C13	106.5 (3)
C4—C3—C2	103.3 (3)	O12—C13—O14	103.8 (2)
C3'—C3—H3	108.6	O12—C13—C16	110.2 (3)
C4—C3—H3	108.6	O14—C13—C16	109.3 (3)
C2—C3—H3	108.6	O12—C13—C15	110.1 (3)
O1—C4—C5	106.6 (3)	O14—C13—C15	110.9 (3)
O1—C4—C3	104.1 (3)	C16—C13—C15	112.2 (3)
C5—C4—C3	115.5 (3)	C2—O14—C13	107.5 (2)
O1—C4—H4	110.1	C13—C15—H15A	109.5
C5—C4—H4	110.1	C13—C15—H15B	109.5
C3—C4—H4	110.1	H15A—C15—H15B	109.5
O9—C5—C4	109.8 (3)	C13—C15—H15C	109.5
O9—C5—C6	104.2 (3)	H15A—C15—H15C	109.5
C4—C5—C6	112.7 (3)	H15B—C15—H15C	109.5
O9—C5—H5	110.0	C13—C16—H16A	109.5
C4—C5—H5	110.0	C13—C16—H16B	109.5
C6—C5—H5	110.0	H16A—C16—H16B	109.5
O7—C6—C5	102.9 (3)	C13—C16—H16C	109.5
O7—C6—H6A	111.2	H16A—C16—H16C	109.5
C5—C6—H6A	111.2	H16B—C16—H16C	109.5
O7—C6—H6B	111.2	N4'—C3'—C3	113.9 (3)
C5—C6—H6B	111.2	N4'—C3'—H3'1	108.8
H6A—C6—H6B	109.1	C3—C3'—H3'1	108.8
C6—O7—C8	106.2 (2)	N4'—C3'—H3'2	108.8
O7—C8—O9	104.8 (3)	C3—C3'—H3'2	108.8
O7—C8—C11	108.5 (3)	H3'1—C3'—H3'2	107.7
O9—C8—C11	109.2 (3)	O6'—N4'—O5'	124.1 (3)
O7—C8—C10	111.7 (3)	O6'—N4'—C3'	116.8 (3)

O9—C8—C10	109.3 (3)	O5'—N4'—C3'	119.1 (3)
C4—O1—C1—O12	82.3 (3)	C6—O7—C8—O9	-32.8 (3)
C4—O1—C1—C2	-30.3 (3)	C6—O7—C8—C11	-149.3 (3)
O1—C1—C2—O14	126.3 (3)	C6—O7—C8—C10	85.4 (3)
O12—C1—C2—O14	9.3 (3)	C4—C5—O9—C8	-115.4 (3)
O1—C1—C2—C3	9.3 (4)	C6—C5—O9—C8	5.6 (4)
O12—C1—C2—C3	-107.7 (3)	O7—C8—O9—C5	16.0 (4)
O14—C2—C3—C3'	23.3 (4)	C11—C8—O9—C5	132.1 (3)
C1—C2—C3—C3'	135.5 (3)	C10—C8—O9—C5	-103.8 (3)
O14—C2—C3—C4	-99.1 (3)	O1—C1—O12—C13	-144.5 (3)
C1—C2—C3—C4	13.1 (3)	C2—C1—O12—C13	-29.2 (3)
C1—O1—C4—C5	161.0 (3)	C1—O12—C13—O14	38.3 (3)
C1—O1—C4—C3	38.5 (3)	C1—O12—C13—C16	155.2 (3)
C3'—C3—C4—O1	-155.8 (3)	C1—O12—C13—C15	-80.4 (3)
C2—C3—C4—O1	-30.8 (3)	C1—C2—O14—C13	13.7 (3)
C3'—C3—C4—C5	87.7 (4)	C3—C2—O14—C13	125.0 (3)
C2—C3—C4—C5	-147.3 (3)	O12—C13—O14—C2	-31.8 (3)
O1—C4—C5—O9	177.9 (3)	C16—C13—O14—C2	-149.4 (3)
C3—C4—C5—O9	-67.0 (4)	C15—C13—O14—C2	86.4 (3)
O1—C4—C5—C6	62.2 (4)	C4—C3—C3'—N4'	-176.0 (3)
C3—C4—C5—C6	177.3 (3)	C2—C3—C3'—N4'	66.2 (4)
O9—C5—C6—O7	-25.0 (3)	C3—C3'—N4'—O6'	-177.3 (3)
C4—C5—C6—O7	94.0 (3)	C3—C3'—N4'—O5'	2.6 (4)
C5—C6—O7—C8	35.7 (3)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3'—H3'2 \cdots O5' ⁱ	0.97	2.53	3.355 (4)	143
C1—H1 \cdots O12 ⁱⁱ	0.98	2.41	3.386 (5)	178
C15—H15A \cdots O6' ⁱⁱⁱ	0.96	2.48	3.433 (5)	174

Symmetry codes: (i) $x-1, y, z$; (ii) $x+1/2, -y+3/2, -z$; (iii) $-x+1, y-1/2, -z+1/2$.