Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,4-Di-*O*-acetyl-2-azidodeoxy-1-*O*-nitro-α-L-fucose

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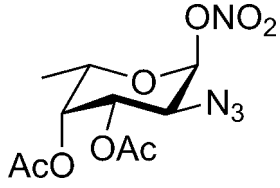
Received 15 June 2007; accepted 6 July 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.084; data-to-parameter ratio = 9.8.

The title compound, $C_{10}H_{14}N_4O_8$, crystallizes with two crystallographically independent molecules in the asymmetric unit, in an arrangement stabilized by a range of non-classical C—H···O hydrogen bonds. The crystals of the α anomer were selectively grown from a mixture of both 1-*O*-nitrates. The compound exhibits the expected $^1C_4$ chair conformation, with the azide group in an equatorial and the nitrate in an axial position.

Related literature

For the synthesis of the title compound, see: Hansen *et al.* (1999); Lehmann *et al.* (1979). For the use of the title compound in aminosugar synthesis, see: Anisuzzaman & Horton (1987); Illarionov *et al.* (1999). For related literature, see: Herbstein (2000); Vainshtein *et al.* (1982).



Experimental

Crystal data

$C_{10}H_{14}N_4O_8$
M_r = 318.25
Orthorhombic, $P2_12_12_1$
a = 8.2169 (6) Å
b = 15.8825 (12) Å
c = 21.7738 (16) Å
V = 2841.6 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 100 (2) K
0.52 × 0.50 × 0.36 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2003)
T_{min} = 0.783, T_{max} = 0.954
25826 measured reflections
3960 independent reflections
3777 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.084
S = 1.05
3960 reflections
403 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10A—H10C···O5A^{i}	0.98	2.42	3.305 (3)	150
C8A—H8A3···O1B^{ii}	0.98	2.52	3.344 (2)	142
C5B—H5B···N4B^{iii}	1.00	2.50	3.403 (3)	150
C4B—H4B···O8B	1.00	2.33	2.706 (2)	101
C3B—H3B···O2A	1.00	2.52	3.515 (2)	174
C3A—H3A···O2B	1.00	2.40	3.390 (2)	173
C2B—H2B···O4B	1.00	2.47	2.838 (2)	101
C2A—H2A···O5A^{i}	1.00	2.54	3.529 (2)	173

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Funding for this research came from the National Institutes of Health (grant No. R15 AI053112-01) and the diffractometer was funded by NSF grant No. 0087210, by Ohio Board of Regents grant No. CAP-491 and by YSU.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2154).

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  doi:10.1107/S160053680703303X

supplementary materials

Acta Cryst. (2007). E**63**, o3484 [**doi:10.1107/S160053680703303X**]

3,4-Di-*O*-acetyl-2-azidodeoxy-1-*O*-nitro-α-L-fucose

D. C. McCutcheon, P. Norris and M. Zeller

Comment

Azidodeoxynitrates such as the tile compound serve as versatile intermediates in the preparation of a variety of aminosugar derivatives. 3,4-Di-*O*-acetyl-2-azidodeoxy-1-*O*-nitro-α-*L*-fucose (I) specifically was used as a precursor for the synthesis of *L*-fucosamine analogs, for example derivatives of the bacterial aminosugar *N*-acetyl-*L*-fucosamine (Anisuzzaman & Horton, 1987), as well as glycosyl phosphates through reaction with caesium dibenzyl phosphate (Illarionov *et al.*, 1999).

(I) was prepared by the reaction of 3,4-di-*O*-acetyl-*L*-fucal (Figure 1, Hansen *et al.*, 1999) with sodium azide and ceric ammonium nitrate in acetone leading to a mixture of 1-*O*-hydroxides and 1-*O*-nitrates with the 2-azidodeoxy-*L*-fucose configuration (Lehmann *et al.*, 1979). The α and β 1-*O*-nitrates can be separated from the 1-*O*-hydroxides by column chromatography on silica gel, and from the mixture of the two nitrates the α-1-*O*-nitrate, namely 3,4-di-*O*-acetyl-2-azidodeoxy-1-*O*-nitro-α-*L*-fucose, was selectively crystallized by vapor diffusion of hexane into a solution of the nitrate mixture in ethyl acetate.

The title compound crystallizes in the orthorhombic chiral space group $P2_12_12_1$ with two crystallographically independent molecules in the asymmetric part of the unit cell. The two independent molecules are chemically identical and both exhibit an almost perfect chair configuration (Figure 2). The rings adopt the $^1C_4$ conformation with the azide groups at C2 in the expected equatorial and the nitrate at C1 in the axial position. The largest deviations between the two independent molecules is found for the azide and the two acetyl groups: the weighted r.m.s. deviation for an overlay of both molecules is 0.24 Å for all non-hydrogen atoms, but drops to only 0.08 Å when the N_3 and OAc groups are omitted from the fit.

The observed arrangement seems to be stabilized by a range of C—H···O and also C—H···N hydrogen bonds. The most significant interactions with C···*X* distances below or close to the sum of the van der Waals radii (Vainshtein *et al.*, 1982) are given in the hydrogen bonding table.

Experimental

Under a nitrogen atmosphere, ceric ammonium nitrate (10.84 g, 19.78 mmol) and sodium azide (1.35 g, 20.76 mmol) were placed in a flame-dried flask equipped with a magnetic stir bar and rubber septum. The reaction vessel was cooled to 258 K (−15 °C) using a dry ice ethylene glycol slush bath. 3,4-Di-*O*-acetyl-*L*-fucal (2.13 g, 9.94 mmol) was dissolved in freshly distilled acetone (50 ml) and transferred *via* cannula to the reaction vessel while maintaining 258 K. Overnight stirring and monitoring by TLC (3:1 petroleum ether-EtOAc) showed consumption of the starting material and the appearance of spots corresponding to the α- and β-anomers of 3,4-di-*O*-acetyl-2-azidodeoxy-1-*O*-nitro-*L*-fucose and the α- and β-anomers of 3,4-di-*O*-acetyl-2-azidodeoxy-*L*-fucose. The reaction mixture was diluted with diethyl ether (40 ml) and washed with H_2O (3 × 20 ml). The organic layer was dried over $MgSO_4$, filtered, and concentrated under reduced pressure. Column chromatography on silica gel (2:1 hexane-ethyl acetate as eluent) provided the mixture of the α- and β-anomers of 3,4-di-*O*-acetyl-2-azidodeoxy-1-*O*-nitro-*L*-fucose in 32% yield and that of 3,4-di-*O*-acetyl-2-azidodeoxy-α,β-*L*-fucose in 64% yield.

Crystals of 3,4-di-*O*-acetyl-2-azidodeoxy-1-*O*-nitro-α-*L*-fucose suitable for X-ray analysis were obtained by vapor diffusion of hexane into a solution of the mixture of the nitrates in ethyl acetate.

Refinement

Friedel pairs were merged prior to refinement and the absolute structure was assigned based on known stereocenters. All hydrogen atoms were placed in calculated positions and were isotropically refined using a riding model, with C—H = 0.97–1.00 Å and with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C) for methyl H atoms. Methyl hydrogen atoms were allowed to rotate to best fit the observed electron density. The e.s.d. values of the cell parameters are taken from the software recognizing that the values are unreasonably small (Herbstein, 2000).

Figures

Fig. 1. Synthesis of the title compound.

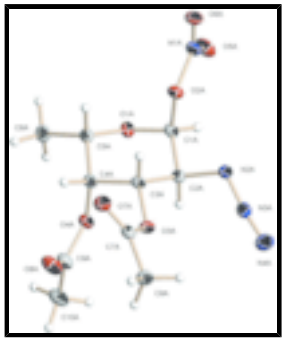


Fig. 2. *ORTEP* presentation of one independent molecule of the title compound with the atom numbering scheme. Anisotropic displacement parameters are at the 50% level.

3,4-Di-*O*-acetyl-2-azidodeoxy-1-*O*-nitro-α-*L*-fucose

Crystal data

$C_{10}H_{14}N_4O_8$	F_{000} = 1328
M_r = 318.25	D_x = 1.488 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 8591 reflections
a = 8.2169 (6) Å	θ = 2.3–30.5°
b = 15.8825 (12) Å	μ = 0.13 mm^{-1}
c = 21.7738 (16) Å	T = 100 (2) K
V = 2841.6 (4) Å^3	Block, colorless
Z = 8	0.52 × 0.50 × 0.36 mm

Data collection

Bruker SMART APEX CCD diffractometer	3960 independent reflections
Radiation source: fine-focus sealed tube	3777 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.057

T = 100(2) K

ω scans

Absorption correction: multi-scan (SADABS in SAINT-Plus; Bruker, 2003)

T_{min} = 0.783, T_{max} = 0.954

25826 measured reflections

θ_{max} = 28.3º

θ_{min} = 1.6º

h = −10→10

k = −21→21

l = −28→28

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.033

$wR(F^2)$ = 0.084

S = 1.05

3960 reflections

403 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0432P)^2 + 0.6271P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 0.27 e Å^{-3}

$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Extinction correction: none

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1A	0.1519 (2)	0.68931 (11)	0.96074 (8)	0.0173 (3)
H1A	0.1697	0.6907	1.0062	0.021*
C2A	0.3138 (2)	0.70045 (11)	0.92821 (8)	0.0165 (3)
H2A	0.3653	0.7545	0.9416	0.020*
C3A	0.2871 (2)	0.70192 (11)	0.85901 (8)	0.0160 (3)
H3A	0.2445	0.6460	0.8452	0.019*
C4A	0.1645 (2)	0.77066 (11)	0.84250 (8)	0.0171 (3)
H4A	0.1394	0.7679	0.7976	0.021*
C5A	0.0083 (2)	0.75793 (11)	0.87924 (8)	0.0178 (3)
H5A	−0.0439	0.7043	0.8654	0.021*
C6A	−0.1136 (2)	0.82860 (12)	0.87166 (9)	0.0234 (4)
H6A1	−0.0730	0.8792	0.8924	0.035*

H6A2	−0.2178	0.8119	0.8898	0.035*
H6A3	−0.1288	0.8405	0.8279	0.035*
C7A	0.4653 (2)	0.68822 (11)	0.77365 (8)	0.0180 (3)
C8A	0.6392 (2)	0.69823 (12)	0.75485 (9)	0.0233 (4)
H8A1	0.6533	0.6775	0.7128	0.035*
H8A2	0.7089	0.6659	0.7827	0.035*
H8A3	0.6694	0.7579	0.7567	0.035*
C9A	0.3093 (3)	0.89460 (12)	0.81195 (9)	0.0230 (4)
C10A	0.3549 (3)	0.98127 (13)	0.83182 (10)	0.0301 (5)
H10A	0.2568	1.0161	0.8347	0.045*
H10B	0.4297	1.0060	0.8018	0.045*
H10C	0.4082	0.9787	0.8720	0.045*
C1B	0.2547 (2)	0.43484 (11)	0.79007 (8)	0.0180 (3)
H1B	0.3018	0.4367	0.7477	0.022*
C2B	0.3935 (2)	0.43329 (11)	0.83734 (8)	0.0178 (3)
H2B	0.4627	0.3826	0.8297	0.021*
C3B	0.3204 (2)	0.42703 (11)	0.90164 (8)	0.0163 (3)
H3B	0.2521	0.4777	0.9105	0.020*
C4B	0.2164 (2)	0.34711 (11)	0.90480 (8)	0.0175 (3)
H4B	0.1664	0.3415	0.9465	0.021*
C5B	0.0837 (2)	0.35172 (11)	0.85600 (8)	0.0178 (3)
H5B	0.0102	0.4000	0.8660	0.021*
C6B	−0.0187 (3)	0.27262 (13)	0.85109 (9)	0.0251 (4)
H6B1	0.0490	0.2262	0.8361	0.038*
H6B2	−0.1086	0.2823	0.8224	0.038*
H6B3	−0.0624	0.2582	0.8916	0.038*
C7B	0.4189 (2)	0.44343 (11)	1.00306 (8)	0.0198 (4)
C8B	0.5676 (3)	0.43756 (15)	1.04241 (10)	0.0304 (4)
H8B1	0.5360	0.4401	1.0858	0.046*
H8B2	0.6407	0.4845	1.0329	0.046*
H8B3	0.6234	0.3842	1.0343	0.046*
C9B	0.3866 (2)	0.23554 (11)	0.94182 (9)	0.0204 (4)
C10B	0.5124 (3)	0.17257 (13)	0.92196 (10)	0.0274 (4)
H10D	0.4929	0.1189	0.9428	0.041*
H10E	0.6210	0.1934	0.9326	0.041*
H10F	0.5054	0.1644	0.8774	0.041*
N1A	−0.0035 (2)	0.56522 (10)	0.98752 (7)	0.0216 (3)
N2A	0.41780 (19)	0.62882 (10)	0.94692 (7)	0.0184 (3)
N3A	0.5655 (2)	0.64117 (10)	0.94132 (7)	0.0206 (3)
N4A	0.7021 (2)	0.64433 (12)	0.93857 (10)	0.0313 (4)
N1B	0.0763 (2)	0.54341 (10)	0.75172 (7)	0.0226 (3)
N2B	0.4937 (2)	0.50950 (10)	0.82922 (8)	0.0209 (3)
N3B	0.6420 (2)	0.49697 (10)	0.83443 (8)	0.0230 (3)
N4B	0.7785 (2)	0.49429 (13)	0.83732 (11)	0.0374 (5)
O1A	0.04098 (16)	0.75143 (8)	0.94464 (6)	0.0181 (3)
O2A	0.09418 (16)	0.60611 (8)	0.94303 (6)	0.0188 (3)
O3A	0.44471 (16)	0.71616 (8)	0.83227 (6)	0.0181 (3)
O4A	0.22958 (17)	0.85305 (8)	0.85736 (6)	0.0191 (3)
O5A	−0.0240 (2)	0.59973 (10)	1.03602 (7)	0.0311 (3)

O6A	−0.05346 (18)	0.49872 (9)	0.96954 (7)	0.0280 (3)
O7A	0.35749 (18)	0.65942 (9)	0.74306 (6)	0.0237 (3)
O8A	0.3355 (2)	0.86470 (9)	0.76178 (7)	0.0311 (3)
O1B	0.15313 (16)	0.36579 (8)	0.79517 (6)	0.0189 (3)
O2B	0.16796 (16)	0.51307 (8)	0.80211 (6)	0.0194 (3)
O3B	0.45529 (16)	0.42301 (8)	0.94370 (6)	0.0191 (3)
O4B	0.32046 (16)	0.27581 (8)	0.89237 (6)	0.0180 (3)
O5B	0.0821 (2)	0.50576 (10)	0.70412 (7)	0.0371 (4)
O6B	0.00131 (18)	0.60629 (9)	0.76422 (7)	0.0265 (3)
O7B	0.28545 (17)	0.46294 (9)	1.01992 (6)	0.0228 (3)
O8B	0.3492 (2)	0.25016 (10)	0.99387 (7)	0.0291 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1A	0.0205 (8)	0.0149 (7)	0.0164 (7)	0.0001 (7)	0.0012 (7)	0.0008 (6)
C2A	0.0171 (8)	0.0150 (7)	0.0172 (8)	−0.0007 (6)	0.0006 (6)	0.0001 (6)
C3A	0.0177 (8)	0.0159 (7)	0.0144 (7)	−0.0017 (7)	0.0033 (6)	−0.0001 (6)
C4A	0.0194 (8)	0.0172 (8)	0.0148 (8)	−0.0010 (7)	0.0007 (6)	0.0003 (6)
C5A	0.0184 (8)	0.0185 (8)	0.0164 (8)	0.0013 (7)	0.0003 (6)	0.0011 (7)
C6A	0.0231 (9)	0.0234 (9)	0.0237 (9)	0.0048 (8)	0.0011 (7)	0.0028 (7)
C7A	0.0216 (9)	0.0152 (7)	0.0172 (8)	−0.0002 (7)	0.0021 (6)	0.0022 (6)
C8A	0.0211 (9)	0.0256 (9)	0.0231 (9)	−0.0024 (8)	0.0039 (7)	0.0001 (7)
C9A	0.0281 (9)	0.0195 (8)	0.0215 (9)	−0.0014 (8)	0.0020 (7)	0.0043 (7)
C10A	0.0432 (12)	0.0219 (9)	0.0252 (10)	−0.0069 (9)	0.0032 (9)	0.0023 (8)
C1B	0.0206 (8)	0.0161 (8)	0.0175 (8)	0.0020 (7)	−0.0007 (7)	0.0003 (6)
C2B	0.0189 (8)	0.0148 (7)	0.0198 (8)	0.0001 (7)	−0.0006 (7)	0.0010 (7)
C3B	0.0158 (8)	0.0161 (7)	0.0169 (8)	0.0012 (7)	−0.0018 (6)	0.0003 (6)
C4B	0.0182 (8)	0.0178 (8)	0.0164 (8)	0.0011 (7)	−0.0005 (6)	−0.0015 (6)
C5B	0.0176 (8)	0.0202 (8)	0.0156 (8)	−0.0003 (7)	0.0000 (6)	−0.0003 (7)
C6B	0.0257 (10)	0.0261 (9)	0.0235 (9)	−0.0068 (8)	−0.0011 (8)	0.0011 (8)
C7B	0.0236 (9)	0.0171 (8)	0.0187 (8)	−0.0035 (7)	−0.0029 (7)	0.0013 (7)
C8B	0.0263 (10)	0.0427 (12)	0.0221 (9)	−0.0012 (9)	−0.0072 (8)	−0.0013 (9)
C9B	0.0224 (8)	0.0167 (8)	0.0220 (9)	−0.0022 (7)	−0.0041 (7)	0.0030 (7)
C10B	0.0314 (10)	0.0229 (9)	0.0278 (10)	0.0079 (8)	−0.0064 (8)	0.0008 (8)
N1A	0.0185 (7)	0.0208 (7)	0.0256 (8)	0.0022 (6)	0.0053 (6)	0.0076 (7)
N2A	0.0169 (7)	0.0165 (7)	0.0218 (7)	−0.0007 (6)	−0.0002 (6)	0.0025 (6)
N3A	0.0225 (8)	0.0175 (7)	0.0218 (7)	0.0019 (6)	0.0006 (6)	0.0008 (6)
N4A	0.0206 (8)	0.0277 (9)	0.0457 (11)	0.0021 (7)	0.0019 (8)	0.0040 (8)
N1B	0.0247 (8)	0.0206 (7)	0.0225 (8)	0.0005 (7)	−0.0058 (6)	0.0022 (6)
N2B	0.0186 (7)	0.0180 (7)	0.0262 (8)	0.0002 (6)	0.0008 (6)	0.0016 (6)
N3B	0.0228 (8)	0.0204 (7)	0.0257 (8)	−0.0015 (7)	−0.0011 (6)	0.0011 (6)
N4B	0.0219 (9)	0.0304 (9)	0.0600 (14)	−0.0021 (8)	−0.0063 (9)	0.0018 (9)
O1A	0.0210 (6)	0.0177 (6)	0.0157 (6)	0.0033 (5)	0.0010 (5)	−0.0003 (5)
O2A	0.0203 (6)	0.0165 (6)	0.0196 (6)	−0.0020 (5)	0.0047 (5)	0.0015 (5)
O3A	0.0174 (6)	0.0191 (6)	0.0178 (6)	−0.0034 (5)	0.0028 (5)	0.0000 (5)
O4A	0.0242 (6)	0.0158 (6)	0.0173 (6)	−0.0021 (5)	0.0019 (5)	0.0013 (5)
O5A	0.0384 (9)	0.0290 (7)	0.0259 (7)	0.0035 (7)	0.0150 (6)	0.0057 (6)

O6A	0.0230 (7)	0.0210 (7)	0.0400 (8)	−0.0047 (6)	0.0017 (6)	0.0063 (6)
O7A	0.0246 (7)	0.0259 (7)	0.0205 (6)	−0.0027 (6)	0.0020 (5)	−0.0024 (5)
O8A	0.0464 (9)	0.0244 (7)	0.0225 (7)	−0.0026 (7)	0.0091 (7)	0.0026 (6)
O1B	0.0229 (6)	0.0174 (6)	0.0162 (6)	−0.0012 (5)	0.0006 (5)	−0.0002 (5)
O2B	0.0234 (6)	0.0170 (6)	0.0179 (6)	0.0033 (5)	−0.0033 (5)	0.0010 (5)
O3B	0.0183 (6)	0.0208 (6)	0.0181 (6)	0.0008 (5)	−0.0033 (5)	−0.0006 (5)
O4B	0.0208 (6)	0.0152 (5)	0.0179 (6)	0.0020 (5)	−0.0022 (5)	0.0003 (5)
O5B	0.0551 (10)	0.0321 (8)	0.0242 (7)	0.0103 (8)	−0.0154 (7)	−0.0057 (6)
O6B	0.0235 (6)	0.0224 (6)	0.0337 (8)	0.0047 (6)	−0.0022 (6)	0.0023 (6)
O7B	0.0240 (7)	0.0242 (7)	0.0201 (6)	−0.0010 (6)	0.0010 (5)	−0.0004 (5)
O8B	0.0382 (8)	0.0292 (7)	0.0198 (7)	0.0051 (7)	−0.0022 (6)	0.0040 (5)

Geometric parameters (Å, °)

C1A—O1A	1.388 (2)	C2B—C3B	1.527 (2)
C1A—O2A	1.456 (2)	C2B—H2B	1.0000
C1A—C2A	1.518 (2)	C3B—O3B	1.440 (2)
C1A—H1A	1.0000	C3B—C4B	1.532 (2)
C2A—N2A	1.480 (2)	C3B—H3B	1.0000
C2A—C3A	1.523 (2)	C4B—O4B	1.445 (2)
C2A—H2A	1.0000	C4B—C5B	1.524 (2)
C3A—O3A	1.438 (2)	C4B—H4B	1.0000
C3A—C4A	1.529 (2)	C5B—O1B	1.459 (2)
C3A—H3A	1.0000	C5B—C6B	1.516 (3)
C4A—O4A	1.450 (2)	C5B—H5B	1.0000
C4A—C5A	1.526 (2)	C6B—H6B1	0.9800
C4A—H4A	1.0000	C6B—H6B2	0.9800
C5A—O1A	1.453 (2)	C6B—H6B3	0.9800
C5A—C6A	1.513 (3)	C7B—O7B	1.197 (2)
C5A—H5A	1.0000	C7B—O3B	1.366 (2)
C6A—H6A1	0.9800	C7B—C8B	1.495 (3)
C6A—H6A2	0.9800	C8B—H8B1	0.9800
C6A—H6A3	0.9800	C8B—H8B2	0.9800
C7A—O7A	1.199 (2)	C8B—H8B3	0.9800
C7A—O3A	1.362 (2)	C9B—O8B	1.197 (2)
C7A—C8A	1.495 (3)	C9B—O4B	1.365 (2)
C8A—H8A1	0.9800	C9B—C10B	1.502 (3)
C8A—H8A2	0.9800	C10B—H10D	0.9800
C8A—H8A3	0.9800	C10B—H10E	0.9800
C9A—O8A	1.210 (2)	C10B—H10F	0.9800
C9A—O4A	1.357 (2)	N1A—O6A	1.199 (2)
C9A—C10A	1.491 (3)	N1A—O5A	1.202 (2)
C10A—H10A	0.9800	N1A—O2A	1.4158 (19)
C10A—H10B	0.9800	N2A—N3A	1.236 (2)
C10A—H10C	0.9800	N3A—N4A	1.125 (2)
C1B—O1B	1.383 (2)	N1B—O5B	1.198 (2)
C1B—O2B	1.456 (2)	N1B—O6B	1.204 (2)
C1B—C2B	1.536 (2)	N1B—O2B	1.4156 (19)
C1B—H1B	1.0000	N2B—N3B	1.240 (2)

C2B—N2B	1.474 (2)	N3B—N4B	1.124 (3)
O1A—C1A—O2A	111.38 (14)	N2B—C2B—H2B	108.9
O1A—C1A—C2A	112.01 (14)	C3B—C2B—H2B	108.9
O2A—C1A—C2A	105.52 (14)	C1B—C2B—H2B	108.9
O1A—C1A—H1A	109.3	O3B—C3B—C2B	106.45 (14)
O2A—C1A—H1A	109.3	O3B—C3B—C4B	111.37 (14)
C2A—C1A—H1A	109.3	C2B—C3B—C4B	108.34 (14)
N2A—C2A—C1A	106.74 (14)	O3B—C3B—H3B	110.2
N2A—C2A—C3A	111.55 (14)	C2B—C3B—H3B	110.2
C1A—C2A—C3A	109.69 (15)	C4B—C3B—H3B	110.2
N2A—C2A—H2A	109.6	O4B—C4B—C5B	109.31 (14)
C1A—C2A—H2A	109.6	O4B—C4B—C3B	108.11 (14)
C3A—C2A—H2A	109.6	C5B—C4B—C3B	109.14 (14)
O3A—C3A—C2A	105.84 (14)	O4B—C4B—H4B	110.1
O3A—C3A—C4A	112.72 (13)	C5B—C4B—H4B	110.1
C2A—C3A—C4A	109.80 (14)	C3B—C4B—H4B	110.1
O3A—C3A—H3A	109.5	O1B—C5B—C6B	106.25 (14)
C2A—C3A—H3A	109.5	O1B—C5B—C4B	111.12 (14)
C4A—C3A—H3A	109.5	C6B—C5B—C4B	113.97 (15)
O4A—C4A—C5A	108.24 (14)	O1B—C5B—H5B	108.5
O4A—C4A—C3A	110.41 (14)	C6B—C5B—H5B	108.5
C5A—C4A—C3A	109.66 (14)	C4B—C5B—H5B	108.5
O4A—C4A—H4A	109.5	C5B—C6B—H6B1	109.5
C5A—C4A—H4A	109.5	C5B—C6B—H6B2	109.5
C3A—C4A—H4A	109.5	H6B1—C6B—H6B2	109.5
O1A—C5A—C6A	106.39 (14)	C5B—C6B—H6B3	109.5
O1A—C5A—C4A	111.57 (14)	H6B1—C6B—H6B3	109.5
C6A—C5A—C4A	113.64 (15)	H6B2—C6B—H6B3	109.5
O1A—C5A—H5A	108.4	O7B—C7B—O3B	123.52 (17)
C6A—C5A—H5A	108.4	O7B—C7B—C8B	126.08 (18)
C4A—C5A—H5A	108.4	O3B—C7B—C8B	110.40 (16)
C5A—C6A—H6A1	109.5	C7B—C8B—H8B1	109.5
C5A—C6A—H6A2	109.5	C7B—C8B—H8B2	109.5
H6A1—C6A—H6A2	109.5	H8B1—C8B—H8B2	109.5
C5A—C6A—H6A3	109.5	C7B—C8B—H8B3	109.5
H6A1—C6A—H6A3	109.5	H8B1—C8B—H8B3	109.5
H6A2—C6A—H6A3	109.5	H8B2—C8B—H8B3	109.5
O7A—C7A—O3A	123.58 (17)	O8B—C9B—O4B	123.61 (18)
O7A—C7A—C8A	126.49 (17)	O8B—C9B—C10B	125.35 (18)
O3A—C7A—C8A	109.92 (16)	O4B—C9B—C10B	111.03 (16)
C7A—C8A—H8A1	109.5	C9B—C10B—H10D	109.5
C7A—C8A—H8A2	109.5	C9B—C10B—H10E	109.5
H8A1—C8A—H8A2	109.5	H10D—C10B—H10E	109.5
C7A—C8A—H8A3	109.5	C9B—C10B—H10F	109.5
H8A1—C8A—H8A3	109.5	H10D—C10B—H10F	109.5
H8A2—C8A—H8A3	109.5	H10E—C10B—H10F	109.5
O8A—C9A—O4A	123.53 (18)	O6A—N1A—O5A	129.84 (17)
O8A—C9A—C10A	125.42 (18)	O6A—N1A—O2A	112.01 (15)
O4A—C9A—C10A	111.03 (16)	O5A—N1A—O2A	118.14 (16)

C9A—C10A—H10A	109.5	N3A—N2A—C2A	114.70 (15)
C9A—C10A—H10B	109.5	N4A—N3A—N2A	172.9 (2)
H10A—C10A—H10B	109.5	O5B—N1B—O6B	129.07 (17)
C9A—C10A—H10C	109.5	O5B—N1B—O2B	118.64 (15)
H10A—C10A—H10C	109.5	O6B—N1B—O2B	112.29 (15)
H10B—C10A—H10C	109.5	N3B—N2B—C2B	113.95 (16)
O1B—C1B—O2B	111.51 (14)	N4B—N3B—N2B	172.6 (2)
O1B—C1B—C2B	112.43 (14)	C1A—O1A—C5A	114.81 (13)
O2B—C1B—C2B	104.85 (14)	N1A—O2A—C1A	114.81 (13)
O1B—C1B—H1B	109.3	C7A—O3A—C3A	116.11 (14)
O2B—C1B—H1B	109.3	C9A—O4A—C4A	117.02 (14)
C2B—C1B—H1B	109.3	C1B—O1B—C5B	115.50 (13)
N2B—C2B—C3B	112.53 (15)	N1B—O2B—C1B	114.29 (13)
N2B—C2B—C1B	108.72 (14)	C7B—O3B—C3B	115.02 (14)
C3B—C2B—C1B	108.86 (15)	C9B—O4B—C4B	117.08 (14)
O1A—C1A—C2A—N2A	−177.50 (14)	C3B—C2B—N2B—N3B	99.33 (19)
O2A—C1A—C2A—N2A	−56.15 (17)	C1B—C2B—N2B—N3B	−140.00 (17)
O1A—C1A—C2A—C3A	−56.49 (19)	O2A—C1A—O1A—C5A	−61.11 (19)
O2A—C1A—C2A—C3A	64.86 (16)	C2A—C1A—O1A—C5A	56.80 (19)
N2A—C2A—C3A—O3A	−64.30 (17)	C6A—C5A—O1A—C1A	−179.81 (14)
C1A—C2A—C3A—O3A	177.64 (13)	C4A—C5A—O1A—C1A	−55.36 (19)
N2A—C2A—C3A—C4A	173.77 (14)	O6A—N1A—O2A—C1A	176.94 (15)
C1A—C2A—C3A—C4A	55.71 (18)	O5A—N1A—O2A—C1A	−3.3 (2)
O3A—C3A—C4A—O4A	−53.14 (18)	O1A—C1A—O2A—N1A	−88.22 (17)
C2A—C3A—C4A—O4A	64.59 (18)	C2A—C1A—O2A—N1A	150.01 (14)
O3A—C3A—C4A—C5A	−172.32 (14)	O7A—C7A—O3A—C3A	5.9 (2)
C2A—C3A—C4A—C5A	−54.59 (19)	C8A—C7A—O3A—C3A	−173.41 (14)
O4A—C4A—C5A—O1A	−67.51 (17)	C2A—C3A—O3A—C7A	155.22 (14)
C3A—C4A—C5A—O1A	53.00 (19)	C4A—C3A—O3A—C7A	−84.75 (18)
O4A—C4A—C5A—C6A	52.77 (19)	O8A—C9A—O4A—C4A	−4.0 (3)
C3A—C4A—C5A—C6A	173.28 (15)	C10A—C9A—O4A—C4A	174.42 (16)
O1B—C1B—C2B—N2B	−179.13 (14)	C5A—C4A—O4A—C9A	−147.08 (16)
O2B—C1B—C2B—N2B	−57.80 (17)	C3A—C4A—O4A—C9A	92.88 (18)
O1B—C1B—C2B—C3B	−56.22 (19)	O2B—C1B—O1B—C5B	−62.93 (18)
O2B—C1B—C2B—C3B	65.10 (17)	C2B—C1B—O1B—C5B	54.51 (19)
N2B—C2B—C3B—O3B	−61.27 (18)	C6B—C5B—O1B—C1B	−178.66 (15)
C1B—C2B—C3B—O3B	178.14 (13)	C4B—C5B—O1B—C1B	−54.18 (19)
N2B—C2B—C3B—C4B	178.85 (14)	O5B—N1B—O2B—C1B	−2.9 (2)
C1B—C2B—C3B—C4B	58.26 (18)	O6B—N1B—O2B—C1B	177.26 (15)
O3B—C3B—C4B—O4B	−56.78 (17)	O1B—C1B—O2B—N1B	−80.51 (17)
C2B—C3B—C4B—O4B	59.97 (17)	C2B—C1B—O2B—N1B	157.57 (14)
O3B—C3B—C4B—C5B	−175.58 (14)	O7B—C7B—O3B—C3B	0.4 (3)
C2B—C3B—C4B—C5B	−58.83 (18)	C8B—C7B—O3B—C3B	−179.70 (15)
O4B—C4B—C5B—O1B	−62.93 (18)	C2B—C3B—O3B—C7B	160.42 (14)
C3B—C4B—C5B—O1B	55.12 (18)	C4B—C3B—O3B—C7B	−81.68 (18)
O4B—C4B—C5B—C6B	57.07 (19)	O8B—C9B—O4B—C4B	8.5 (3)
C3B—C4B—C5B—C6B	175.12 (15)	C10B—C9B—O4B—C4B	−170.63 (15)
C1A—C2A—N2A—N3A	−157.79 (16)	C5B—C4B—O4B—C9B	−147.23 (15)
C3A—C2A—N2A—N3A	82.4 (2)	C3B—C4B—O4B—C9B	94.07 (17)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10A—H10C···O5A^{i}	0.98	2.42	3.305 (3)	150
C8A—H8A3···O1B^{ii}	0.98	2.52	3.344 (2)	142
C5B—H5B···N4B^{iii}	1.00	2.50	3.403 (3)	150
C4B—H4B···O8B	1.00	2.33	2.706 (2)	101
C3B—H3B···O2A	1.00	2.52	3.515 (2)	174
C3A—H3A···O2B	1.00	2.40	3.390 (2)	173
C2B—H2B···O4B	1.00	2.47	2.838 (2)	101
C2A—H2A···O5A^{i}	1.00	2.54	3.529 (2)	173

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

Fig. 1

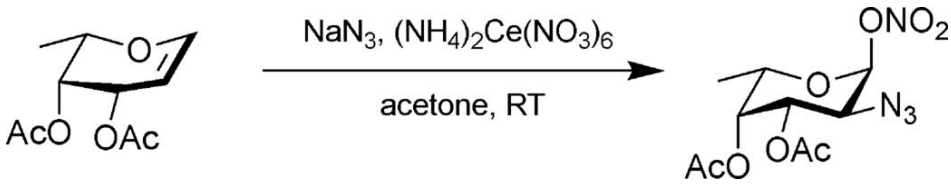
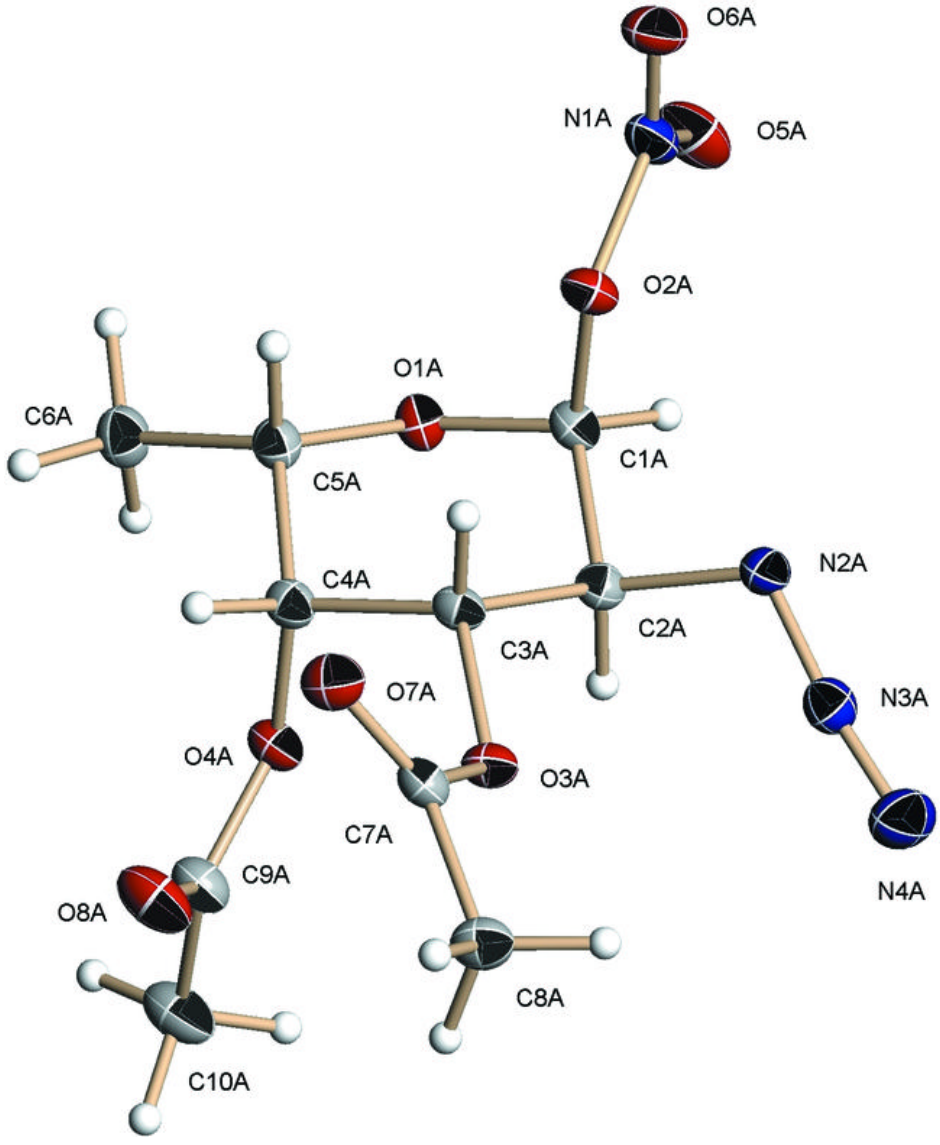


Fig. 2


O6A
N1A
O5A
O2A
O1A
C6A
C5A
C1A
N2A
C4A
C2A
C3A
O7A
N3A
O4A
O3A
C7A
N4A
C9A
O8A
C8A
C10A