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N-Benzyl-3,5-dideoxy-3,5-imino-1,2-*O*-isopropylidene-β-L-lyxofuranose

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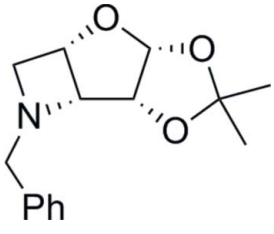
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.003 Å; R factor = 0.033; wR factor = 0.087; data-to-parameter ratio = 9.5.

X-ray crystallography confirmed the formation, structure and relative stereochemistry of the title compound, $C_{15}H_{19}NO_3$, which contains a sterically congested four-membered azetidine ring system. The absolute configuration was determined by the use of L-arabinose as the starting material.

Related literature

For related literature on azetidines, see: Krämer *et al.* (1997); Michaud *et al.* (1997*a*,*b*); Dekaris & Reissig (2010); Soengas *et al.* (2011); Jenkinson *et al.* (2011); Lenagh-Snow *et al.* (2011, 2012); Lee *et al.* (2012). For related literature on iminosugars, see: Asano *et al.* (2000); Watson *et al.* (2001). For details of the cryostat, see: Cosier & Glazer (1986). For details of hydrogen refinement, see: Cooper *et al.* (2010). For references to the Chebychev polynomial, see: Prince (1982); Watkin (1994).



Experimental

Crystal data

$C_{15}H_{19}NO_3$
M_r = 261.32
Monoclinic, $P2_1$
a = 9.1674 (2) Å
b = 5.7551 (1) Å
c = 13.1112 (3) Å
β = 106.9544 (8)°
V = 661.67 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 150 K
0.24 × 0.23 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.94, T_{max} = 0.99
13110 measured reflections
1638 independent reflections
1544 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.087
S = 0.94
1638 reflections
172 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5500).

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Asano, N., Nash, R. J., Molyneuxl, R. J. & Fleet, G. W. J. (2000). *Tetrahedron Asymmetry*, **11**, 1645–1680.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Cooper, R. I., Thompson, A. L. & Watkin, D. J. (2010). *J. Appl. Cryst.* **43**, 1100–1107.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Dekaris, V. & Reissig, H.-U. (2010). *Synlett*, pp. 42–46.
Jenkinson, S. F., Lenagh-Snow, G. M. J., Fleet, G. W. J. & Thompson, A. L. (2011). *Acta Cryst.* E**67**, o2452.
Krämer, B., Franz, T., Picasso, S., Pruschek, P. & Jäger, V. (1997). *Synlett*, pp. 95–97.
Lee, J. C., Francis, S., Dutta, D., Gupta, V., Yang, Y., Zhu, J.-Y., Tash, J. S., Schonbrunn, E. & Georg, G. I. (2012). *J. Org. Chem.* **77**, 3082–3098.
Lenagh-Snow, G. M. J., Araujo, N., Jenkinson, S. F., Martinez, R. F., Shimada, Y., Yu, C.-Y., Kato, A. & Fleet, G. W. J. (2012). *Org. Lett.* **14**, 2142–2145.
Lenagh-Snow, G. M. J., Araujo, N., Jenkinson, S. F., Rutherford, C., Nakagawa, S., Kato, A., Yu, C.-Y., Weymouth-Wilson, A. C. & Fleet, G. W. J. (2011). *Org. Lett.* **13**, 5834–5837.
Michaud, T., Chanet-Ray, J., Chou, S. & Gelas, J. (1997*a*). *Carbohydr. Res.* **299**, 253–269.
Michaud, T., Chanet-Ray, J., Chou, S. & Gelas, J. (1997*b*). *Carbohydr. Res.* **303**, 123–127.
Nonius (2001). *COLLECT*. 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.
Prince, E. (1982). In *Mathematical Techniques in Crystallography and Materials Science*. New York: Springer-Verlag.
Soengas, R. G., Segade, Y., Jiménez, C. & Rodríguez, J. (2011). *Tetrahedron*, **67**, 2617–2622.
Watkin, D. (1994). *Acta Cryst.* A**50**, 411–437.
Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, Oxford, England.
Watson, A. A., Fleet, G. W. J., Asano, N., Molyneux, R. J. & Nash, R. J. (2001). *Phytochemistry*, **56**, 265–295.

supporting information

Acta Cryst. (2012). E**68**, o2410 [https://doi.org/10.1107/S1600536812030656]

N-Benzyl-3,5-dideoxy-3,5-imino-1,2-*O*-isopropylidene-*β*-L-lyxofuranose

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S1. Comment

Azetidines (Michaud *et al.*, 1997*a*; Michaud *et al.*, 1997*b*; Dekaris & Reissig, 2010; Soengas *et al.*, 2011) are a relatively unstudied class of iminosugars (Asano *et al.*, 2000; Watson *et al.*, 2001, Michaud *et al.*, 1997*a*; Michaud *et al.*, 1997*b*; Dekaris & Reissig, 2010; Soengas *et al.*, 2011) but initial results (Krämer *et al.*, 1997; Lee *et al.*, 2012) have shown some interesting biological activity.

Azetidine formation can be achieved by the double displacement of ditriflates with amines (Jenkinson *et al.*, 2011; Lenagh-Snow *et al.*, 2011; Lenagh-Snow *et al.*, 2012). X-Ray crystallography confirmed the structure and relative stereochemistry of the formation of the title compound **3** (Fig. 1) from the displacement of a 3,5-*O*-ditriflate **2** with benzylamine. The absolute stereochemistry was determined by the use of L-arabinose as the starting material.

The five membered rings adopt envelope conformations with O7 and C10 out of the plane, and the azetidine ring adopts a puckered conformation (Fig. 2, Fig. 3).

S2. Experimental

The title compound was recrystallized from cyclohexane/pentane. $[\alpha]_D^{25}$ +76.0 (*c* 0.50 in $CHCl_3$); m.p. 337–339 K.

S3. Refinement

In the absence of significant anomalous scattering, Friedel pairs were merged and the absolute configuration was assigned from the starting material.

The H atoms were all located in a difference map, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98 Å) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints (Cooper *et al.*, 2010).

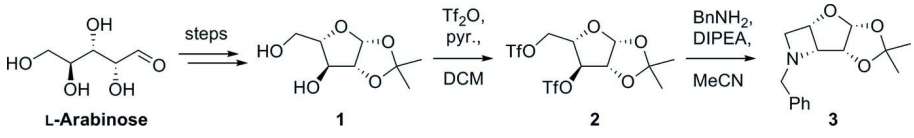

Figure 1

Synthetic Scheme.

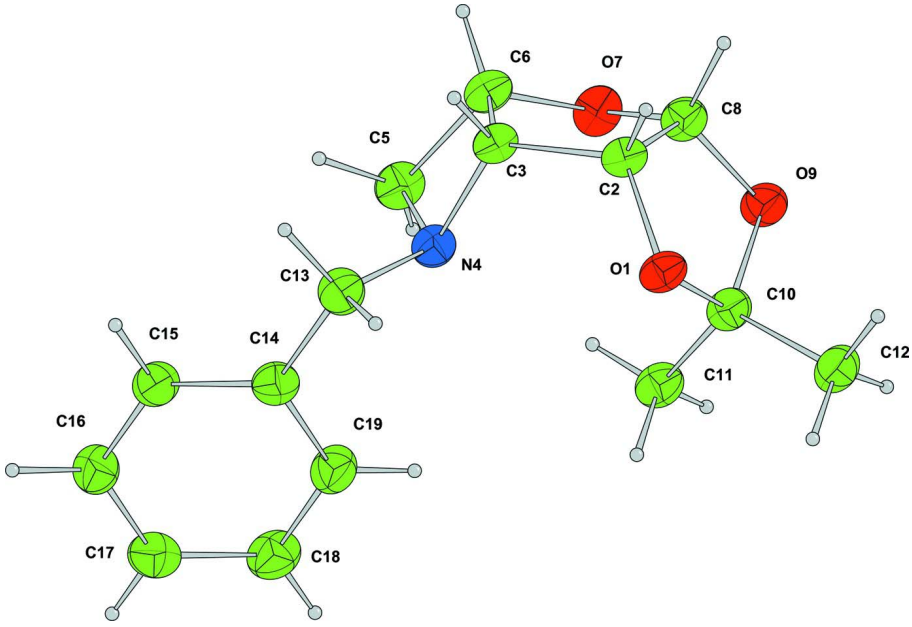

Figure 2

The title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

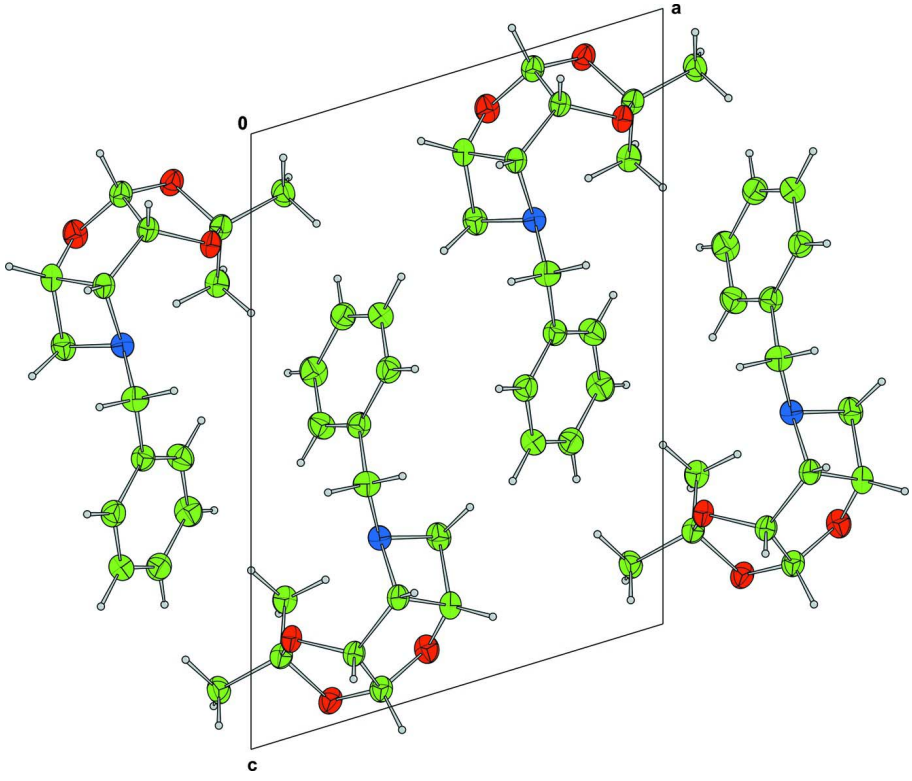

Figure 3

Packing diagram of the title compound projected along the *b*-axis.

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Crystal data

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$M_r = 261.32$
Monoclinic, $P2_1$
Hall symbol: P 2yb
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$b = 5.7551$ (1) Å
$c = 13.1112$ (3) Å
$\beta = 106.9544$ (8)°
$V = 661.67$ (2) Å^3
$Z = 2$
$F(000) = 280$
$D_x = 1.312$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1627 reflections
$\theta = 5$–27°
$\mu = 0.09$ mm^{-1}
$T = 150$ K
Plate, colourless
0.24 × 0.23 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.94$, $T_{max} = 0.99$
13110 measured reflections
1638 independent reflections
1544 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 27.4°$, $\theta_{min} = 5.4°$
$h = -11 \rightarrow 11$
$k = -7 \rightarrow 6$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.087$
$S = 0.94$
1638 reflections
172 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
H-atom parameters constrained
Method, part 1, Chebychev polynomial, (Watkin, 1994, *P*rince, 1982) [*w*eight] = 1.0/[A_0*T_0(x) + A_1*T_1(x) ··· + A_{n-1}]*T_{n-1}(x)] where A_i are the Chebychev coefficients listed below and x = *F* /*F*max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta*F*/6*sigma*F*)^2]^2 A_i are: 22.1 34.0 17.6 5.07
$(\Delta/\sigma)_{max} = 0.0003$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems open-flow nitrogen cryostat (Cosier & Glazer, 1986) with a nominal stability of 0.1 K.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.09793 (13)	0.5625 (2)	0.84062 (10)	0.0269
C2	0.25050 (17)	0.6293 (3)	0.89568 (13)	0.0260
C3	0.35745 (18)	0.6618 (3)	0.82587 (13)	0.0262
N4	0.31206 (15)	0.5481 (3)	0.72006 (11)	0.0264
C5	0.45568 (19)	0.4116 (4)	0.74729 (15)	0.0318
C6	0.48402 (19)	0.4783 (3)	0.86494 (14)	0.0292
O7	0.42651 (14)	0.3113 (3)	0.92411 (10)	0.0319
C8	0.31601 (19)	0.4200 (3)	0.96665 (14)	0.0284

O9	0.19278 (13)	0.2728 (3)	0.96037 (10)	0.0313
C10	0.07349 (18)	0.3246 (3)	0.86343 (14)	0.0274
C11	0.0820 (2)	0.1650 (3)	0.77364 (14)	0.0306
C12	−0.07829 (19)	0.3058 (4)	0.88768 (15)	0.0333
C13	0.2813 (2)	0.6967 (3)	0.62624 (14)	0.0309
C14	0.2617 (2)	0.5526 (4)	0.52659 (14)	0.0288
C15	0.3343 (2)	0.6152 (4)	0.45124 (14)	0.0320
C16	0.3150 (2)	0.4828 (4)	0.35950 (14)	0.0353
C17	0.2236 (2)	0.2866 (4)	0.34202 (15)	0.0346
C18	0.1518 (2)	0.2208 (4)	0.41731 (16)	0.0379
C19	0.1714 (2)	0.3523 (4)	0.50899 (15)	0.0343
H21	0.2489	0.7722	0.9365	0.0300*
H31	0.3962	0.8232	0.8266	0.0305*
H51	0.4416	0.2480	0.7305	0.0388*
H52	0.5313	0.4779	0.7148	0.0374*
H61	0.5871	0.5325	0.9042	0.0348*
H81	0.3666	0.4639	1.0430	0.0337*
H112	0.0678	0.0054	0.7931	0.0457*
H113	0.0002	0.2059	0.7092	0.0453*
H111	0.1807	0.1782	0.7612	0.0454*
H121	−0.0909	0.1489	0.9112	0.0506*
H123	−0.1589	0.3444	0.8220	0.0494*
H122	−0.0782	0.4156	0.9454	0.0507*
H132	0.1871	0.7872	0.6208	0.0373*
H131	0.3679	0.8067	0.6310	0.0372*
H151	0.3977	0.7507	0.4630	0.0386*
H161	0.3646	0.5290	0.3064	0.0422*
H171	0.2100	0.2000	0.2782	0.0423*
H181	0.0896	0.0854	0.4067	0.0458*
H191	0.1208	0.3077	0.5589	0.0420*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0214 (5)	0.0238 (6)	0.0331 (6)	0.0007 (5)	0.0041 (4)	0.0028 (5)
C2	0.0234 (7)	0.0239 (8)	0.0291 (7)	−0.0006 (6)	0.0051 (6)	−0.0014 (7)
C3	0.0234 (7)	0.0242 (8)	0.0299 (7)	−0.0018 (6)	0.0063 (6)	−0.0014 (6)
N4	0.0255 (6)	0.0246 (7)	0.0293 (7)	−0.0002 (6)	0.0081 (5)	−0.0014 (6)
C5	0.0287 (8)	0.0290 (9)	0.0385 (9)	0.0023 (7)	0.0108 (7)	−0.0040 (8)
C6	0.0225 (7)	0.0289 (9)	0.0349 (8)	−0.0002 (7)	0.0063 (6)	−0.0003 (7)
O7	0.0293 (6)	0.0270 (6)	0.0393 (6)	0.0054 (5)	0.0100 (5)	0.0060 (6)
C8	0.0251 (7)	0.0274 (9)	0.0313 (8)	0.0014 (7)	0.0058 (6)	0.0036 (7)
O9	0.0264 (6)	0.0333 (7)	0.0311 (6)	−0.0017 (5)	0.0034 (5)	0.0078 (5)
C10	0.0248 (7)	0.0257 (8)	0.0299 (8)	−0.0007 (7)	0.0054 (6)	0.0049 (7)
C11	0.0291 (8)	0.0254 (8)	0.0361 (8)	−0.0019 (7)	0.0077 (7)	0.0013 (7)
C12	0.0264 (7)	0.0366 (10)	0.0376 (8)	−0.0030 (8)	0.0105 (6)	0.0037 (8)
C13	0.0365 (8)	0.0249 (8)	0.0318 (8)	−0.0035 (8)	0.0106 (7)	−0.0007 (7)
C14	0.0268 (7)	0.0279 (9)	0.0307 (8)	0.0010 (7)	0.0066 (6)	−0.0015 (7)

C15	0.0289 (8)	0.0330 (10)	0.0326 (8)	−0.0028 (8)	0.0069 (6)	0.0022 (7)
C16	0.0307 (8)	0.0430 (12)	0.0337 (9)	0.0009 (8)	0.0116 (7)	0.0003 (8)
C17	0.0306 (8)	0.0374 (10)	0.0342 (8)	0.0042 (8)	0.0070 (7)	−0.0071 (8)
C18	0.0374 (9)	0.0322 (10)	0.0447 (10)	−0.0062 (8)	0.0129 (8)	−0.0080 (9)
C19	0.0357 (9)	0.0317 (10)	0.0379 (9)	−0.0066 (8)	0.0147 (7)	−0.0034 (8)

Geometric parameters (Å, º)

O1—C2	1.4271 (19)	C11—H112	0.972
O1—C10	1.433 (2)	C11—H113	0.981
C2—C3	1.535 (2)	C11—H111	0.967
C2—C8	1.534 (2)	C12—H121	0.972
C2—H21	0.984	C12—H123	0.983
C3—N4	1.480 (2)	C12—H122	0.985
C3—C6	1.542 (2)	C13—C14	1.513 (2)
C3—H31	0.994	C13—H132	0.993
N4—C5	1.484 (2)	C13—H131	1.003
N4—C13	1.457 (2)	C14—C15	1.390 (2)
C5—C6	1.536 (2)	C14—C19	1.399 (3)
C5—H51	0.967	C15—C16	1.391 (3)
C5—H52	0.990	C15—H151	0.957
C6—O7	1.429 (2)	C16—C17	1.385 (3)
C6—H61	0.986	C16—H161	0.974
O7—C8	1.435 (2)	C17—C18	1.389 (3)
C8—O9	1.395 (2)	C17—H171	0.950
C8—H81	1.005	C18—C19	1.387 (3)
O9—C10	1.4456 (19)	C18—H181	0.952
C10—C11	1.513 (3)	C19—H191	0.941
C10—C12	1.519 (2)		
C2—O1—C10	109.99 (13)	O9—C10—C12	107.79 (14)
O1—C2—C3	115.67 (13)	O1—C10—C12	108.68 (15)
O1—C2—C8	104.34 (14)	C11—C10—C12	112.21 (15)
C3—C2—C8	104.59 (14)	C10—C11—H112	109.1
O1—C2—H21	109.4	C10—C11—H113	108.9
C3—C2—H21	109.7	H112—C11—H113	109.0
C8—C2—H21	113.1	C10—C11—H111	110.3
C2—C3—N4	116.83 (13)	H112—C11—H111	108.8
C2—C3—C6	105.56 (14)	H113—C11—H111	110.7
N4—C3—C6	89.27 (13)	C10—C12—H121	109.5
C2—C3—H31	113.4	C10—C12—H123	107.6
N4—C3—H31	115.1	H121—C12—H123	111.0
C6—C3—H31	113.9	C10—C12—H122	108.6
C3—N4—C5	91.22 (12)	H121—C12—H122	109.0
C3—N4—C13	117.65 (15)	H123—C12—H122	111.1
C5—N4—C13	116.97 (14)	N4—C13—C14	110.62 (16)
N4—C5—C6	89.30 (13)	N4—C13—H132	108.6
N4—C5—H51	114.2	C14—C13—H132	110.1

C6—C5—H51	116.3	N4—C13—H131	111.3
N4—C5—H52	112.1	C14—C13—H131	107.0
C6—C5—H52	113.7	H132—C13—H131	109.2
H51—C5—H52	109.9	C13—C14—C15	120.71 (17)
C3—C6—C5	86.95 (13)	C13—C14—C19	120.65 (16)
C3—C6—O7	106.25 (13)	C15—C14—C19	118.64 (17)
C5—C6—O7	113.33 (15)	C14—C15—C16	120.39 (18)
C3—C6—H61	118.1	C14—C15—H151	119.4
C5—C6—H61	117.3	C16—C15—H151	120.3
O7—C6—H61	112.3	C15—C16—C17	120.55 (17)
C6—O7—C8	109.39 (14)	C15—C16—H161	120.0
C2—C8—O7	107.62 (14)	C17—C16—H161	119.4
C2—C8—O9	105.91 (13)	C16—C17—C18	119.59 (18)
O7—C8—O9	111.33 (16)	C16—C17—H171	119.4
C2—C8—H81	113.1	C18—C17—H171	121.0
O7—C8—H81	108.9	C17—C18—C19	119.92 (19)
O9—C8—H81	110.0	C17—C18—H181	120.5
C8—O9—C10	108.58 (13)	C19—C18—H181	119.6
O9—C10—O1	104.85 (14)	C14—C19—C18	120.89 (17)
O9—C10—C11	111.18 (15)	C14—C19—H191	119.8
O1—C10—C11	111.77 (14)	C18—C19—H191	119.3

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H111···N4	0.97	2.58	3.266 (3)	128