Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(4*R*)-4-(2-Allyl-2*H*-1,2,3-triazol-4-yl)-1,2-*O*-isopropylidene-L-threose

Sarah F. Jenkinson,[a]* Daniel Best,[a] Francis X. Wilson,[b] George W. J. Fleet[a] and David J. Watkin[c]

[a]Department of Organic Chemistry, Chemistry Research Laboratory, Department of Chemistry, University of Oxford, Oxford OX1 3TA, England, [b]Summit plc, 91 Milton Park, Abingdon, Oxfordshire OX14 4RY, England, and [c]Department of Chemical Crystallography, Chemistry Research Laboratory, Department of Chemistry, University of Oxford, Oxford OX1 3TA, England
Correspondence e-mail: sarah.jenkinson@chem.no.ac.uk

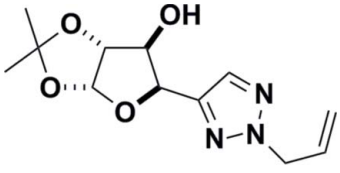
Received 29 October 2008; accepted 6 November 2008

Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.004 Å; R factor = 0.041; wR factor = 0.098; data-to-parameter ratio = 8.9.

X-ray crystallography unequivocally confirmed the structure of the title compound, $C_{12}H_{17}N_3O_4$, as (4*R*)-4-(2-allyl-2*H*-1,2,3-triazol-4-yl)-1,2-*O*-isopropylidene-L-threose. The absolute configuration was determined by the use of D-glucorono-3,6-lactone as the starting material. The crystal structure consists of hydrogen-bonded chains of molecules running parallel to the *a* axis. There are no unusual packing features.

Related literature

For related background information on the biotechnological interconversion of monosaccharides and other sugars, see: Izumori (2002, 2006); Granstrom *et al.* (2004); Yoshihara *et al.* (2008); Booth *et al.* (2008); Jenkinson, Booth, Gullapalli *et al.* (2008); Jenkinson, Booth, Yoshihara *et al.* (2008); Gullapalli *et al.* (2007); Jenkinson, Booth, Best *et al.* (2008). For related literature, see: Görbitz (1999).



Experimental

Crystal data

$C_{12}H_{17}N_3O_4$
M_r = 267.28
Orthorhombic, $P2_12_12_1$
a = 5.3959 (2) Å
b = 9.6233 (3) Å
c = 25.4532 (9) Å
V = 1321.69 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 150 K
0.30 × 0.20 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.82, T_{max} = 1.00
(expected range = 0.817–0.997)
9466 measured reflections
1528 independent reflections
1194 reflections with $I > 2\sigma(I)$
R_{int} = 0.096

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.098
S = 0.93
1528 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O11—H111···O8^{i}	0.88	1.95	2.822 (4)	170

Symmetry code: (i) $x+1, y, z$.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*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*.

We thank the Oxford University Crystallography Service for access to equipment.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2725).

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Booth, K. V., Jenkinson, S. F., Fleet, G. W. J., Gullapalli, P., Yoshihara, A., Izumori, K. & Watkin, D. J. (2008). *Acta Cryst.* E**64**, o1385.
Görbitz, C. H. (1999). *Acta Cryst.* B**55**, 1090–1098.
Granstrom, T. B., Takata, G., Tokuda, M. & Izumori, K. (2004). *J. Biosci. Bioeng.* **97**, 89–94.
Gullapalli, P., Shiji, T., Rao, D., Yoshihara, A., Morimoto, K., Takata, G., Fleet, G. W. J. & Izumori, K. (2007). *Tetrahedron Asymmetry*, **18**, 1995–2000.
Izumori, K. (2002). *Naturwissenschaften*, **89**, 120–124.
Izumori, K. (2006). *J. Biotechnol.* **124**, 717–722.
Jenkinson, S. F., Booth, K. V., Best, D., Fleet, G. W. J. & Watkin, D. J. (2008). *Acta Cryst.* E**64**, o2011–o2012.
Jenkinson, S. F., Booth, K. V., Gullapalli, P., Morimoto, K., Izumori, K., Fleet, G. W. J. & Watkin, D. J. (2008). *Acta Cryst.* E**64**, o1705–o1706.
Jenkinson, S. F., Booth, K. V., Yoshihara, A., Morimoto, K., Fleet, G. W. J., Izumori, K. & Watkin, D. J. (2008). *Acta Cryst.* E**64**, o1429.
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.
Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*, Chemical Crystallography Laboratory, Oxford, England.
Yoshihara, A., Haraguchi, S., Gullapalli, P., Rao, D., Morimoto, K., Takata, G., Jones, N., Jenkinson, S. F., Wormald, M. R., Dwek, R. A., Fleet, G. W. J. & Izumori, K. (2008). *Tetrahedron Asymmetry*, **19**, 739–745.

supporting information

Acta Cryst. (2008). E**64**, o2361 [doi:10.1107/S1600536808036416]

(4*R*)-4-(2-Allyl-2*H*-1,2,3-triazol-4-yl)-1,2-*O*-isopropylidene-L-threose

Sarah F. Jenkinson, Daniel Best, Francis X. Wilson, George W. J. Fleet and David J. Watkin

S1. Comment

The process for the biotechnological interconversion of monosaccharides developed by Izumori (Izumori, 2002; Izumori, 2006; Granstrom *et al.*, 2004), has been seen to be generally applicable to other sugar derivatives such as 1-deoxy sugars (Yoshihara *et al.*, 2008; Booth *et al.* 2008; Jenkinson, Booth, Gullapalli *et al.*, 2008; Jenkinson, Booth, Yoshihara *et al.*, 2008; Gullapalli *et al.*, 2007). To evaluate the applicability of this process to 2-deoxy sugars and their derivatives a variety of carbon chain extension reactions were investigated, for example, addition of lithium *tert*-butyl acetate to sugar lactones (Jenkinson, Booth, Best *et al.*, 2008) or addition of allyl magnesium bromide to an aldose.

Reaction of lactol **1** (Fig. 1) with 2.5 equivalents of allyl magnesium bromide generated a single isolable product along with recovered starting material. X-ray crystallography identified the compound as 4*R*-4-(2-allyl-2*H*-1,2,3-triazole-4-yl)-1,2-*O*-isopropylidene-L-threose **2** (Fig. 2) rather than the anticipated addition product **3**. The crystal structure was seen to consist of alternating chains of hydrogen-bonded molecules running parallel to the *a*-axis (Fig. 3).Only classic intermolecular hydrogen bonding has been considered. The absolute configuration was determined from the starting material.

S2. Experimental

The title compound was recrystallized by vapour diffusion from a mixture of diethyl ether and cyclohexane: m.p. 361–364 K; $[\alpha]_D^{25}$ -13.9 (*c*, 0.69 in $CHCl_3$).

S3. Refinement

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

The relatively large ratio of minimum to maximum corrections applied in the multiscan process (1:1.22) reflect changes in the illuminated volume of the crystal. Changes in illuminated volume were kept to a minimum, and were taken into account (Görbitz, 1999) by the multi-scan inter-frame scaling (*DENZO*/*SCALEPACK*, Otwinowski & Minor, 1997).

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, O—H = 0.82 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.

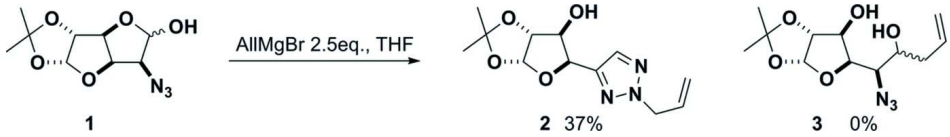


Figure 1

Synthetic Scheme

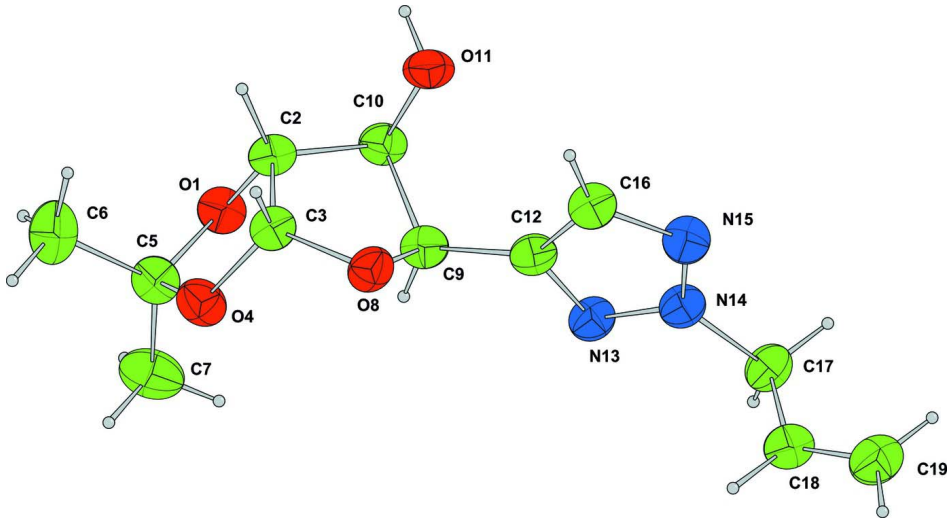


Figure 2

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

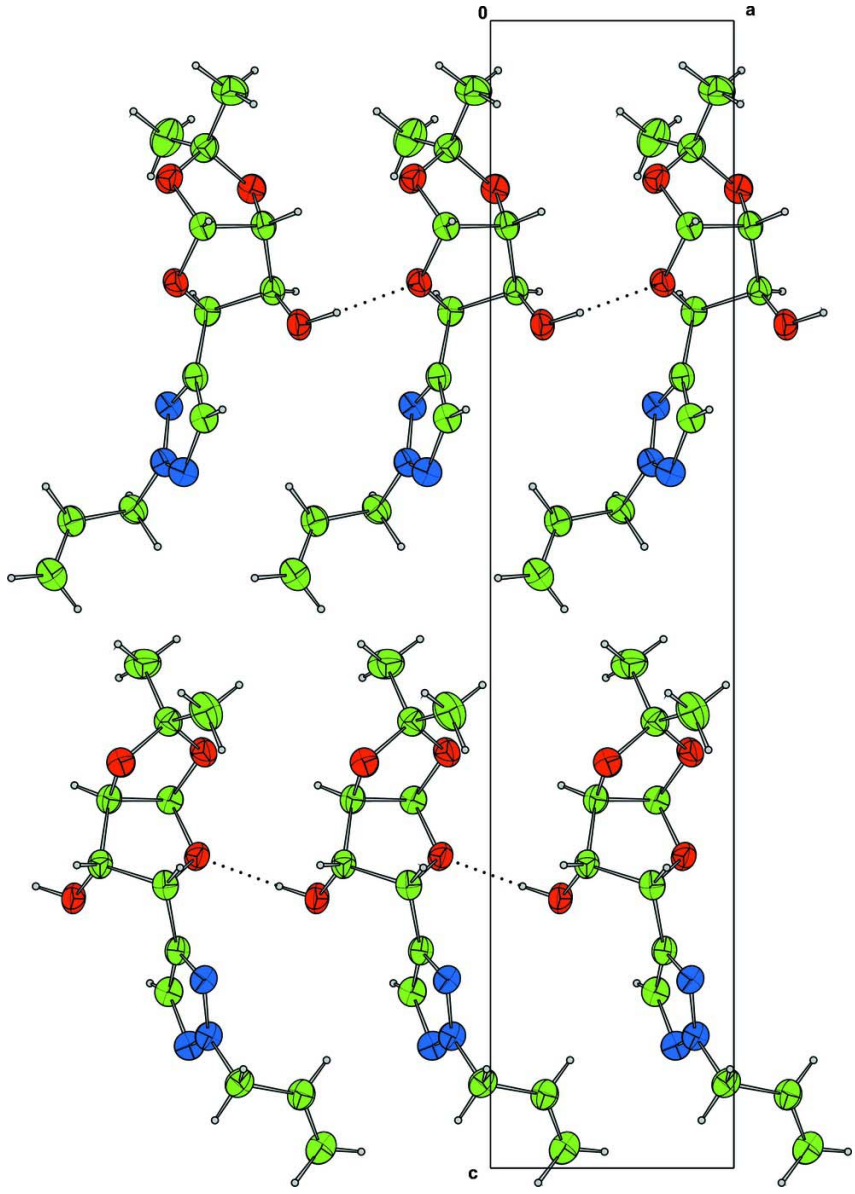


Figure 3

Packing diagram for the title compound projected along the *b*-axis. Hydrogen bonds are indicated by dotted lines.

(4*R*)-4-(2-Allyl-2*H*-1,2,3-triazol-4-yl)-1,2-*O*- isopropylidene-L-threose

Crystal data

$C_{12}H_{17}N_3O_4$
$M_r = 267.28$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.3959\ (2)$ Å
$b = 9.6233\ (3)$ Å
$c = 25.4532\ (9)$ Å
$V = 1321.69\ (8)$ Å^3
$Z = 4$
$F(000) = 568$
$D_x = 1.343$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1500 reflections
$\theta = 5$–$26°$
$\mu = 0.10$ mm^{-1}
$T = 150$ K
Plate, colourless
0.30 × 0.20 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.82, T_{max} = 1.00
9466 measured reflections
1528 independent reflections
1194 reflections with $I > 2\sigma(I)$
R_{int} = 0.096
θ_{max} = 26.0°, θ_{min} = 5.3°
$h = -6 \rightarrow 6$
$k = -11 \rightarrow 11$
$l = -30 \rightarrow 31$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.098
S = 0.93
1528 reflections
172 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.59P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max}$ = 0.000120
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.0189 (4)	0.60546 (19)	0.14662 (7)	0.0395
C2	1.0670 (5)	0.7244 (3)	0.17865 (11)	0.0366
C3	0.8174 (6)	0.8008 (3)	0.17938 (10)	0.0374
O4	0.6798 (4)	0.7427 (2)	0.13781 (7)	0.0436
C5	0.8248 (6)	0.6390 (3)	0.11107 (11)	0.0407
C6	0.9294 (7)	0.7022 (4)	0.06094 (12)	0.0579
C7	0.6697 (7)	0.5117 (4)	0.10209 (15)	0.0608
O8	0.7063 (4)	0.7727 (2)	0.22853 (7)	0.0394
C9	0.8362 (6)	0.6572 (3)	0.25347 (11)	0.0371
C10	1.1041 (5)	0.6779 (3)	0.23535 (11)	0.0371
O11	1.2131 (4)	0.7866 (2)	0.26506 (8)	0.0425
C12	0.7862 (6)	0.6641 (3)	0.31060 (11)	0.0359
N13	0.6792 (5)	0.5578 (2)	0.33594 (9)	0.0372
N14	0.6586 (5)	0.6032 (2)	0.38514 (9)	0.0372
N15	0.7386 (5)	0.7336 (2)	0.39351 (9)	0.0406
C16	0.8223 (6)	0.7724 (3)	0.34651 (11)	0.0396
C17	0.5321 (6)	0.5246 (3)	0.42598 (12)	0.0404
C18	0.2777 (6)	0.5809 (3)	0.43621 (12)	0.0439
C19	0.1964 (7)	0.6160 (3)	0.48272 (12)	0.0499
H21	1.2090	0.7805	0.1665	0.0468*
H31	0.8420	0.9045	0.1748	0.0468*
H61	0.7897	0.7321	0.0390	0.0897*
H62	1.0308	0.6330	0.0433	0.0901*
H63	1.0262	0.7815	0.0727	0.0903*
H73	0.5330	0.5360	0.0790	0.0953*
H72	0.7699	0.4402	0.0859	0.0956*

H71	0.6044	0.4809	0.1356	0.0950*
H91	0.7757	0.5674	0.2382	0.0502*
H101	1.1990	0.5896	0.2360	0.0485*
H161	0.8989	0.8595	0.3389	0.0499*
H171	0.5156	0.4288	0.4138	0.0506*
H172	0.6324	0.5300	0.4584	0.0502*
H181	0.1730	0.5914	0.4060	0.0573*
H192	0.0314	0.6524	0.4857	0.0646*
H191	0.3038	0.6046	0.5128	0.0648*
H111	1.3679	0.7927	0.2545	0.0633*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0413 (12)	0.0331 (10)	0.0440 (11)	0.0027 (10)	−0.0005 (10)	−0.0048 (9)
C2	0.0328 (14)	0.0335 (15)	0.0434 (16)	−0.0013 (13)	0.0025 (13)	−0.0016 (13)
C3	0.0372 (14)	0.0345 (14)	0.0406 (15)	0.0007 (14)	0.0016 (14)	0.0007 (13)
O4	0.0369 (10)	0.0492 (12)	0.0447 (11)	0.0076 (11)	−0.0044 (10)	−0.0066 (10)
C5	0.0407 (16)	0.0378 (15)	0.0436 (16)	0.0052 (15)	−0.0022 (15)	−0.0015 (13)
C6	0.070 (2)	0.058 (2)	0.0457 (18)	0.0116 (19)	0.0039 (18)	0.0051 (17)
C7	0.057 (2)	0.0484 (19)	0.077 (2)	−0.007 (2)	−0.013 (2)	−0.0080 (18)
O8	0.0321 (10)	0.0450 (11)	0.0411 (10)	0.0061 (10)	0.0042 (9)	0.0053 (9)
C9	0.0349 (15)	0.0313 (14)	0.0451 (17)	0.0007 (13)	−0.0013 (14)	0.0035 (12)
C10	0.0315 (15)	0.0367 (15)	0.0430 (16)	0.0022 (12)	0.0012 (13)	−0.0064 (14)
O11	0.0292 (10)	0.0498 (11)	0.0486 (11)	−0.0048 (10)	0.0023 (9)	−0.0075 (10)
C12	0.0324 (14)	0.0326 (13)	0.0427 (15)	0.0006 (13)	0.0015 (14)	0.0005 (12)
N13	0.0381 (13)	0.0328 (12)	0.0408 (13)	−0.0013 (12)	0.0040 (12)	−0.0010 (10)
N14	0.0380 (13)	0.0320 (12)	0.0415 (13)	−0.0020 (12)	0.0021 (12)	0.0009 (11)
N15	0.0456 (14)	0.0334 (12)	0.0429 (13)	−0.0006 (12)	0.0012 (11)	−0.0013 (11)
C16	0.0408 (15)	0.0332 (14)	0.0447 (16)	0.0013 (15)	0.0017 (14)	0.0009 (13)
C17	0.0420 (16)	0.0359 (15)	0.0433 (17)	0.0005 (14)	0.0062 (14)	0.0027 (14)
C18	0.0396 (17)	0.0435 (16)	0.0486 (17)	−0.0038 (15)	0.0027 (15)	0.0003 (15)
C19	0.0482 (18)	0.0462 (17)	0.0552 (19)	−0.0032 (18)	0.0095 (18)	−0.0042 (15)

Geometric parameters (Å, º)

O1—C2	1.429 (3)	C9—C12	1.481 (4)
O1—C5	1.421 (4)	C9—H91	1.003
C2—C3	1.535 (4)	C10—O11	1.419 (3)
C2—C10	1.524 (4)	C10—H101	0.992
C2—H21	0.987	O11—H111	0.879
C3—O4	1.409 (3)	C12—N13	1.340 (3)
C3—O8	1.414 (3)	C12—C16	1.400 (4)
C3—H31	1.013	N13—N14	1.331 (3)
O4—C5	1.439 (3)	N14—N15	1.344 (3)
C5—C6	1.522 (4)	N14—C17	1.456 (4)
C5—C7	1.501 (4)	N15—C16	1.332 (4)
C6—H61	0.982	C16—H161	0.954

C6—H62	0.972	C17—C18	1.498 (4)
C6—H63	0.973	C17—H171	0.977
C7—H73	0.972	C17—H172	0.988
C7—H72	0.968	C18—C19	1.307 (4)
C7—H71	0.969	C18—H181	0.959
O8—C9	1.459 (3)	C19—H192	0.960
C9—C10	1.531 (4)	C19—H191	0.967

C2—O1—C5	108.4 (2)	C10—C9—C12	117.5 (3)
O1—C2—C3	103.4 (2)	O8—C9—H91	109.4
O1—C2—C10	109.2 (2)	C10—C9—H91	107.6
C3—C2—C10	104.2 (2)	C12—C9—H91	111.1
O1—C2—H21	113.6	C9—C10—C2	101.5 (2)
C3—C2—H21	115.0	C9—C10—O11	109.1 (2)
C10—C2—H21	110.8	C2—C10—O11	110.0 (2)
C2—C3—O4	105.3 (2)	C9—C10—H101	111.8
C2—C3—O8	106.9 (2)	C2—C10—H101	109.6
O4—C3—O8	111.4 (2)	O11—C10—H101	114.1
C2—C3—H31	110.9	C10—O11—H111	106.3
O4—C3—H31	112.0	C9—C12—N13	121.1 (2)
O8—C3—H31	110.2	C9—C12—C16	130.5 (3)
C3—O4—C5	110.1 (2)	N13—C12—C16	108.3 (2)
O4—C5—O1	104.9 (2)	C12—N13—N14	103.8 (2)
O4—C5—C6	108.8 (2)	N13—N14—N15	115.4 (2)
O1—C5—C6	110.6 (3)	N13—N14—C17	122.7 (2)
O4—C5—C7	109.5 (3)	N15—N14—C17	121.5 (2)
O1—C5—C7	108.8 (2)	N14—N15—C16	103.2 (2)
C6—C5—C7	113.9 (3)	C12—C16—N15	109.3 (3)
C5—C6—H61	108.0	C12—C16—H161	125.6
C5—C6—H62	108.8	N15—C16—H161	125.1
H61—C6—H62	111.7	N14—C17—C18	111.5 (2)
C5—C6—H63	104.7	N14—C17—H171	107.9
H61—C6—H63	110.9	C18—C17—H171	108.2
H62—C6—H63	112.2	N14—C17—H172	108.2
C5—C7—H73	108.6	C18—C17—H172	109.7
C5—C7—H72	109.5	H171—C17—H172	111.4
H73—C7—H72	109.7	C17—C18—C19	123.9 (3)
C5—C7—H71	108.6	C17—C18—H181	116.0
H73—C7—H71	109.2	C19—C18—H181	120.0
H72—C7—H71	111.1	C18—C19—H192	118.6
C3—O8—C9	109.1 (2)	C18—C19—H191	119.2
O8—C9—C10	102.9 (2)	H192—C19—H191	122.2
O8—C9—C12	107.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H161···O1^{i}	0.95	2.44	3.322 (4)	154

C17—H171···O4^{ii}	0.98	2.46	3.362 (4)	154
O11—H111···O8^{iii}	0.88	1.95	2.822 (4)	170

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