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# 2-Azido-2-deoxy-3,4-*O*-isopropylidene-2-*C*-methyl-D-talono-1,5-lactone

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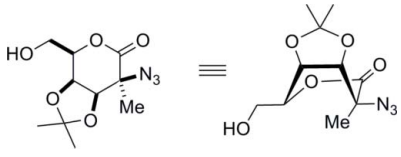
Key indicators: single-crystal X-ray study; $T$ = 150 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.038; $wR$ factor = 0.087; data-to-parameter ratio = 10.0.

The relative stereochemistry of the title compound, $C_{10}H_{15}N_3O_5$, was confirmed by the crystal structure determination. The absolute configuration was determined from the use of D-lyxonolactone as the starting material. The six-membered ring adopts a boat conformation with the larger azide group, rather than the methyl group, in the bowsprit position. In the crystal structure, a bifurcated intermolecular O—H···O/O—H···N hydrogen bond links molecules into chains running parallel to the *b* axis.

## Related literature

For carbohydrates as chirons, see: Lichtenthaler & Peters (2004); Fechter *et al.* (1999); Fleet (1989). For branched sugars and their use as chirons, see: Rao *et al.* (2008); Jones *et al.* (2008); Booth *et al.* (2008); Hotchkiss, Kato *et al.* (2007); da Cruz *et al.* (2008); Soengas *et al.* (2005). For the structures of similar sugars, see: Chesterton *et al.* (2006); Booth *et al.* (2007); Hotchkiss, Jenkinson *et al.* (2007); Baird *et al.* (1987); Bruce *et al.* (1990); Punzo *et al.* (2005). For the extinction correction, see: Larson (1970).




## Experimental

### Crystal data

$C_{10}H_{15}N_3O_5$
$M_r$ = 257.25
Orthorhombic, $P2_12_12_1$
$a$ = 5.9481 (3) Å
$b$ = 13.3427 (7) Å
$c$ = 15.6351 (9) Å
$V$ = 1240.86 (12) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 150 K
0.20 × 0.15 × 0.05 mm

### Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min}$ = 0.89, $T_{max}$ = 0.99
10775 measured reflections
1647 independent reflections
1170 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.077

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.087
$S$ = 0.88
1647 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.45 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O15—H151···O1$^{i}$ | 0.84 | 2.14 | 2.930 (4) | 157 |
| O15—H151···N7$^{i}$ | 0.84 | 2.52 | 3.072 (4) | 125 |

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5031).

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# supporting information

*Acta Cryst.* (2010). E**66**, o1221–o1222 [https://doi.org/10.1107/S160053681001500X]

## 2-Azido-2-deoxy-3,4-*O*-isopropylidene-2-*C*-methyl-D-talono-1,5-lactone

**Sarah F. Jenkinson, Ni Dai, George W. J. Fleet and David J. Watkin**

### S1. Comment

Carbohydrates are a diverse set of chirons for the synthesis of complex amino acids and iminosugars (Lichtenthaler & Peters, 2004; Fechter et al., 1999; Fleet, 1989). 2-C-Methyl branched sugars constitute a class of rare sugars with chemotherapeutic potential (Rao et al., 2008; Jones et al., 2008; Booth et al., 2008) and can be used as building blocks in the synthesis of biologically active compounds (da Cruz et al., 2008; Hotchkiss, Kato et al., 2007; Soengas et al., 2005).

The azidolactone 3 (Fig. 1) would be a key intermediate for the synethsis of branched pyrrolidines, piperidines and prolines derived from D-lyxonolactone. Nucleophilic displacement of a triflate leaving group at the tertiary centre by azide was confirmed by X-ray crystallography to have proceeded with overall inversion of configuration (Booth et al. 2007; Hotchkiss, Jenkinson et al. 2007). The 6-membered lactone ring adopts a boat conformation, as is common with 3,4-O-isopropylidene-1,5-lactones (Baird et al., 1987; Bruce et al., 1990; Punzo et al., 2005), with the larger azide group, rather than the methyl, in the bowsprit position (Fig. 2). The absolute configuration was determined from the use of D-lyxonolactone as the starting material. As is common with these materials the azide is non linear [N7 - N8 - N9 = 172.4 (3) °] (Chesterton et al., 2006), with the anisotropic atomic displacement parameter of the central atom lowered with respect to its neighbours. The compound exists as hydrogen bonded chains of molecules running parallel to the b-axis (Fig. 3). The hydrogen bond is bifurcated. Only classical hydrogen bonding is considered.

### S2. Experimental

The title compound was recrystallised by slow evaporation from a mixture of diethyl ether and cyclohexane: m.p. 397-403 K, $[\alpha]_D^{25}$ +112.4 (*c*, 1.145 in $CHCl_3$).

### S3. Refinement

In the absence of significant anomalous scattering, Friedel pairs were merged and the absolute configuration was assigned from the use of D-lyonolactone as 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, 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.

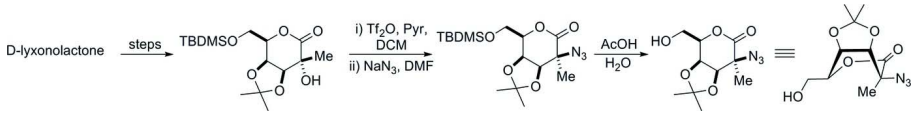


**Figure 1**
Synthetic Scheme

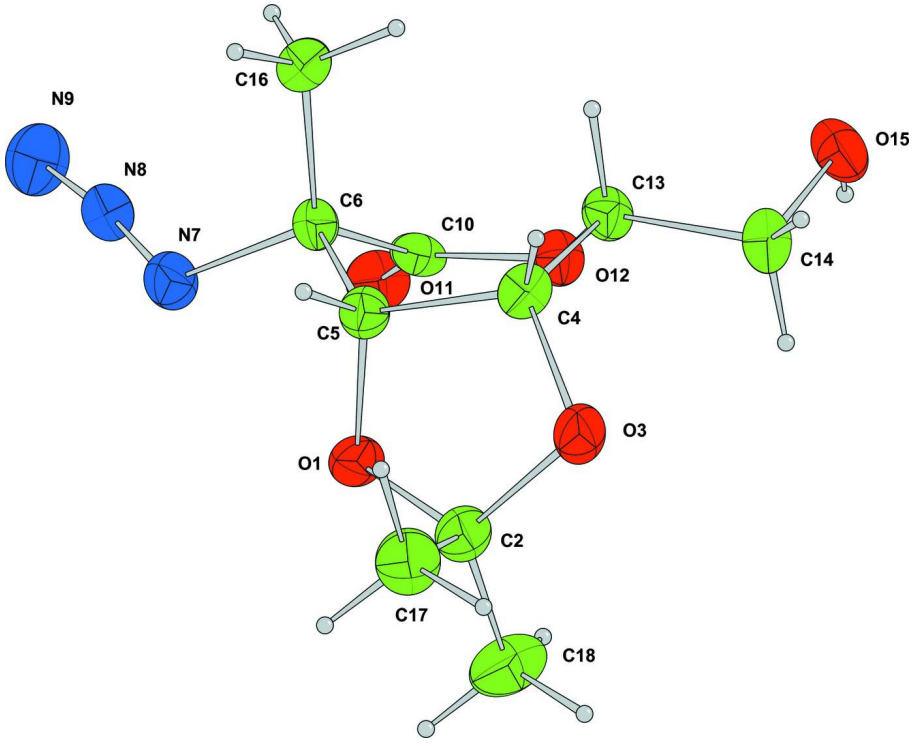


**Figure 2**

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

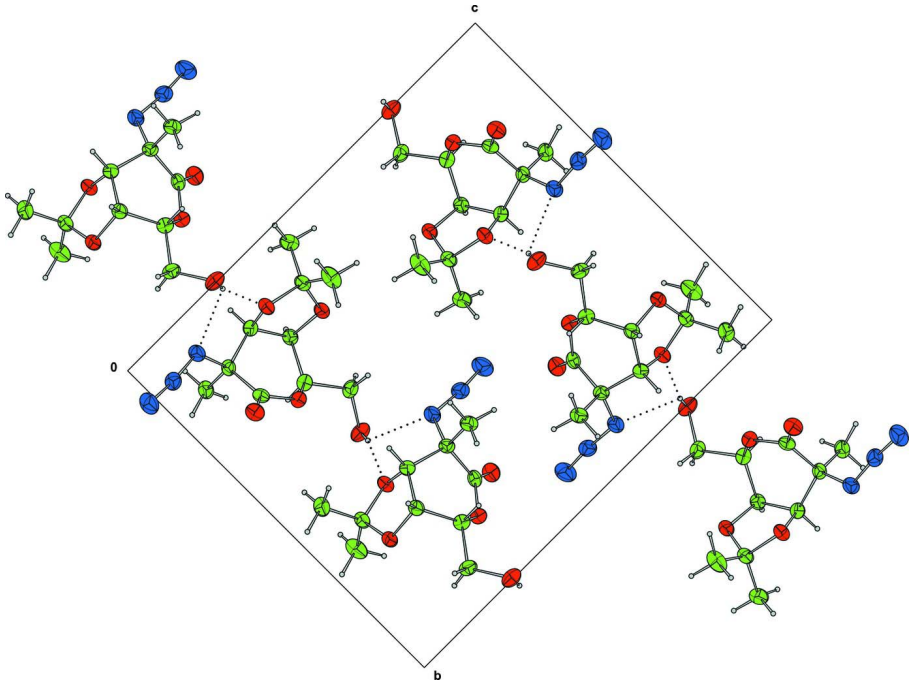


**Figure 3**

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

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*Crystal data*

$C_{10}H_{15}N_3O_5$
$M_r = 257.25$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.9481\ (3)$ Å
$b = 13.3427\ (7)$ Å
$c = 15.6351\ (9)$ Å
$V = 1240.86\ (12)$ Å$^3$
$Z = 4$
$F(000) = 544$
$D_x = 1.377$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1637 reflections
$\theta = 5$–$27°$
$\mu = 0.11$ mm$^{-1}$
$T = 150$ K
Plate, colourless
0.20 × 0.15 × 0.05 mm

*Data collection*

Nonius KappaCCD diffractometer
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.89$, $T_{max} = 0.99$
10775 measured reflections
1647 independent reflections
1170 reflections with $I > 2\sigma(I)$
$R_{int} = 0.077$
$\theta_{max} = 27.5°$, $\theta_{min} = 5.2°$
$h = -7 \rightarrow 7$
$k = -17 \rightarrow 17$
$l = -20 \rightarrow 20$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.087$
$S = 0.88$
1647 reflections
164 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.05P)^2 + 0.16P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.000278$
$\Delta\rho_{max} = 0.53$ e Å$^{-3}$
$\Delta\rho_{min} = -0.45$ e Å$^{-3}$
Extinction correction: Larson (1970), Equation 22
Extinction coefficient: 460 (60)

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.4977 (3) | 0.87439 (12) | 0.79200 (10) | 0.0276 |
| C2 | 0.5736 (5) | 0.85325 (19) | 0.87769 (16) | 0.0297 |
| O3 | 0.7326 (3) | 0.77432 (13) | 0.86551 (10) | 0.0334 |
| C4 | 0.8307 (4) | 0.78267 (17) | 0.78210 (15) | 0.0258 |
| C5 | 0.6901 (4) | 0.86484 (17) | 0.73767 (14) | 0.0250 |
| C6 | 0.6110 (4) | 0.83522 (18) | 0.64929 (15) | 0.0247 |
| N7 | 0.4436 (4) | 0.91275 (16) | 0.62475 (14) | 0.0317 |
| N8 | 0.3742 (4) | 0.90581 (16) | 0.55031 (15) | 0.0313 |
| N9 | 0.2976 (4) | 0.90888 (18) | 0.48383 (15) | 0.0443 |
| C10 | 0.4914 (4) | 0.73333 (18) | 0.65603 (15) | 0.0243 |
| O11 | 0.3123 (3) | 0.71606 (13) | 0.62348 (11) | 0.0323 |
| O12 | 0.5913 (3) | 0.66364 (12) | 0.70449 (11) | 0.0256 |
| C13 | 0.8169 (4) | 0.68186 (17) | 0.73740 (16) | 0.0250 |

| | | | | |
|---|---|---|---|---|
| C14 | 0.8716 (5) | 0.59403 (17) | 0.79413 (17) | 0.0309 |
| O15 | 0.8866 (3) | 0.50433 (11) | 0.74599 (11) | 0.0351 |
| C16 | 0.8056 (4) | 0.83600 (19) | 0.58502 (16) | 0.0303 |
| C17 | 0.6857 (5) | 0.9437 (2) | 0.91665 (18) | 0.0385 |
| C18 | 0.3762 (5) | 0.8142 (2) | 0.92680 (19) | 0.0459 |
| H41 | 0.9905 | 0.8032 | 0.7872 | 0.0311* |
| H51 | 0.7740 | 0.9284 | 0.7350 | 0.0310* |
| H131 | 0.9235 | 0.6813 | 0.6888 | 0.0288* |
| H141 | 1.0180 | 0.6075 | 0.8209 | 0.0398* |
| H142 | 0.7552 | 0.5873 | 0.8388 | 0.0391* |
| H161 | 0.7461 | 0.8167 | 0.5292 | 0.0461* |
| H162 | 0.8707 | 0.9027 | 0.5818 | 0.0463* |
| H163 | 0.9219 | 0.7893 | 0.6024 | 0.0460* |
| H172 | 0.7391 | 0.9258 | 0.9730 | 0.0598* |
| H171 | 0.5743 | 0.9972 | 0.9206 | 0.0603* |
| H173 | 0.8113 | 0.9635 | 0.8797 | 0.0603* |
| H182 | 0.4260 | 0.7957 | 0.9845 | 0.0690* |
| H181 | 0.2604 | 0.8655 | 0.9297 | 0.0694* |
| H183 | 0.3174 | 0.7559 | 0.8975 | 0.0688* |
| H151 | 0.7591 | 0.4778 | 0.7453 | 0.0532* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0272 (9) | 0.0348 (9) | 0.0207 (8) | 0.0036 (8) | −0.0008 (8) | 0.0009 (7) |
| C2 | 0.0358 (14) | 0.0323 (13) | 0.0209 (12) | 0.0025 (12) | −0.0027 (11) | −0.0011 (12) |
| O3 | 0.0480 (11) | 0.0305 (9) | 0.0217 (9) | 0.0089 (9) | −0.0031 (8) | 0.0004 (8) |
| C4 | 0.0254 (13) | 0.0278 (12) | 0.0241 (13) | −0.0041 (11) | −0.0031 (10) | 0.0007 (11) |
| C5 | 0.0249 (12) | 0.0250 (12) | 0.0252 (12) | 0.0006 (10) | −0.0006 (11) | 0.0016 (11) |
| C6 | 0.0253 (12) | 0.0255 (12) | 0.0233 (13) | 0.0057 (11) | −0.0011 (11) | 0.0031 (10) |
| N7 | 0.0361 (12) | 0.0337 (11) | 0.0253 (11) | 0.0090 (10) | −0.0035 (10) | −0.0003 (10) |
| N8 | 0.0309 (12) | 0.0300 (11) | 0.0330 (13) | 0.0066 (10) | 0.0013 (11) | 0.0030 (11) |
| N9 | 0.0446 (14) | 0.0544 (16) | 0.0340 (14) | 0.0089 (13) | −0.0105 (12) | 0.0052 (12) |
| C10 | 0.0210 (12) | 0.0297 (13) | 0.0221 (11) | 0.0039 (11) | 0.0023 (11) | −0.0047 (11) |
| O11 | 0.0255 (9) | 0.0391 (10) | 0.0322 (10) | −0.0029 (9) | −0.0047 (8) | −0.0022 (9) |
| O12 | 0.0228 (8) | 0.0246 (8) | 0.0293 (9) | −0.0005 (7) | −0.0023 (7) | 0.0012 (8) |
| C13 | 0.0192 (11) | 0.0267 (12) | 0.0292 (14) | 0.0008 (10) | −0.0021 (11) | 0.0020 (11) |
| C14 | 0.0327 (14) | 0.0247 (12) | 0.0351 (14) | 0.0018 (12) | −0.0044 (12) | 0.0045 (12) |
| O15 | 0.0297 (9) | 0.0248 (9) | 0.0509 (12) | 0.0035 (8) | 0.0037 (9) | 0.0003 (9) |
| C16 | 0.0318 (13) | 0.0317 (13) | 0.0274 (13) | 0.0010 (12) | 0.0036 (11) | 0.0049 (11) |
| C17 | 0.0484 (17) | 0.0374 (15) | 0.0297 (14) | 0.0015 (14) | −0.0060 (14) | −0.0059 (12) |
| C18 | 0.0446 (17) | 0.063 (2) | 0.0300 (16) | −0.0052 (15) | 0.0033 (13) | 0.0041 (15) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C2 | 1.442 (3) | C10—O12 | 1.338 (3) |
| O1—C5 | 1.431 (3) | O12—C13 | 1.458 (3) |
| C2—O3 | 1.428 (3) | C13—C14 | 1.505 (3) |

| | | | |
|---|---|---|---|
| C2—C17 | 1.508 (4) | C13—H131 | 0.989 |
| C2—C18 | 1.496 (4) | C14—O15 | 1.417 (3) |
| O3—C4 | 1.433 (3) | C14—H141 | 0.983 |
| C4—C5 | 1.544 (3) | C14—H142 | 0.987 |
| C4—C13 | 1.518 (3) | O15—H151 | 0.837 |
| C4—H41 | 0.993 | C16—H161 | 0.977 |
| C5—C6 | 1.512 (3) | C16—H162 | 0.972 |
| C5—H51 | 0.985 | C16—H163 | 0.969 |
| C6—N7 | 1.486 (3) | C17—H172 | 0.967 |
| C6—C10 | 1.538 (3) | C17—H171 | 0.976 |
| C6—C16 | 1.533 (3) | C17—H173 | 0.981 |
| N7—N8 | 1.238 (3) | C18—H182 | 0.981 |
| N8—N9 | 1.136 (3) | C18—H181 | 0.972 |
| C10—O11 | 1.203 (3) | C18—H183 | 0.969 |

| | | | |
|---|---|---|---|
| C2—O1—C5 | 106.47 (18) | C4—C13—O12 | 111.08 (19) |
| O1—C2—O3 | 103.17 (18) | C4—C13—C14 | 114.0 (2) |
| O1—C2—C17 | 110.9 (2) | O12—C13—C14 | 106.09 (19) |
| O3—C2—C17 | 110.6 (2) | C4—C13—H131 | 109.0 |
| O1—C2—C18 | 107.4 (2) | O12—C13—H131 | 108.6 |
| O3—C2—C18 | 109.4 (2) | C14—C13—H131 | 108.0 |
| C17—C2—C18 | 114.7 (2) | C13—C14—O15 | 111.0 (2) |
| C2—O3—C4 | 109.49 (17) | C13—C14—H141 | 107.5 |
| O3—C4—C5 | 104.14 (19) | O15—C14—H141 | 109.0 |
| O3—C4—C13 | 109.17 (18) | C13—C14—H142 | 109.6 |
| C5—C4—C13 | 113.14 (19) | O15—C14—H142 | 110.1 |
| O3—C4—H41 | 109.8 | H141—C14—H142 | 109.7 |
| C5—C4—H41 | 111.0 | C14—O15—H151 | 107.9 |
| C13—C4—H41 | 109.5 | C6—C16—H161 | 108.1 |
| C4—C5—O1 | 103.26 (17) | C6—C16—H162 | 110.0 |
| C4—C5—C6 | 113.20 (19) | H161—C16—H162 | 109.8 |
| O1—C5—C6 | 108.46 (18) | C6—C16—H163 | 110.5 |
| C4—C5—H51 | 110.9 | H161—C16—H163 | 109.9 |
| O1—C5—H51 | 110.7 | H162—C16—H163 | 108.6 |
| C6—C5—H51 | 110.1 | C2—C17—H172 | 108.4 |
| C5—C6—N7 | 105.21 (19) | C2—C17—H171 | 108.1 |
| C5—C6—C10 | 108.20 (19) | H172—C17—H171 | 110.3 |
| N7—C6—C10 | 108.84 (18) | C2—C17—H173 | 108.3 |
| C5—C6—C16 | 111.2 (2) | H172—C17—H173 | 110.7 |
| N7—C6—C16 | 109.39 (19) | H171—C17—H173 | 111.0 |
| C10—C6—C16 | 113.6 (2) | C2—C18—H182 | 108.8 |
| C6—N7—N8 | 114.5 (2) | C2—C18—H181 | 109.6 |
| N7—N8—N9 | 172.4 (3) | H182—C18—H181 | 110.4 |
| C6—C10—O11 | 123.4 (2) | C2—C18—H183 | 108.6 |
| C6—C10—O12 | 116.6 (2) | H182—C18—H183 | 110.0 |
| O11—C10—O12 | 120.0 (2) | H181—C18—H183 | 109.4 |
| C10—O12—C13 | 119.50 (19) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C5—H51···O15$^{i}$ | 0.99 | 2.28 | 3.141 (4) | 146 |
| C13—H131···O11$^{ii}$ | 0.99 | 2.57 | 3.473 (4) | 152 |
| C16—H161···O11$^{iii}$ | 0.98 | 2.46 | 3.333 (4) | 149 |
| C16—H163···O11$^{ii}$ | 0.97 | 2.54 | 3.465 (4) | 159 |
| O15—H151···O1$^{iv}$ | 0.84 | 2.14 | 2.930 (4) | 157 |
| O15—H151···N7$^{iv}$ | 0.84 | 2.52 | 3.072 (4) | 125 |

Symmetry codes: (i) −*x*+2, *y*+1/2, −*z*+3/2; (ii) *x*+1, *y*, *z*; (iii) *x*+1/2, −*y*+3/2, −*z*+1; (iv) −*x*+1, *y*−1/2, −*z*+3/2.