α-Lactose monohydrate: a redetermination at 150 K

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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 = 0.079
Data-to-parameter ratio = 8.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the monohydrate of α-4-(β-D-galactopyranosido)-D-glucopyranose, more commonly known as α-lactose monohydrate, $C_{12}H_{22}O_{11}\cdot H_2O$, has been previously studied by single-crystal diffraction at *ca* 296 K [Beevers & Hansen (1971). *Acta Cryst.* B**27**, 1323–1325; Fries *et al.* (1971). *Acta Cryst.* B**27**, 994–1005; Noordik *et al.* (1984). *Z. Kristallogr.* **168**, 59–65]. This redetermination at low temperature [150 (2) K] shows improved precision of geometry. Graph-set analysis of the hydrogen-bonding motifs is presented for the first time.
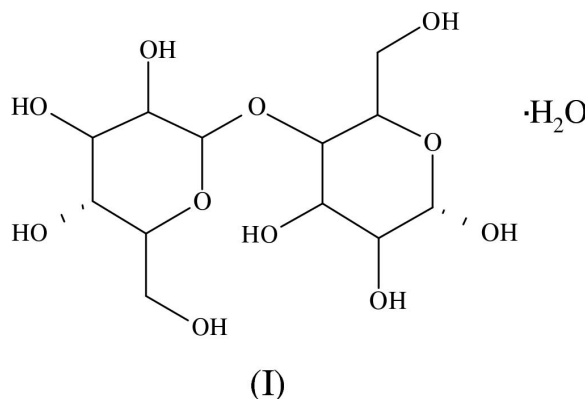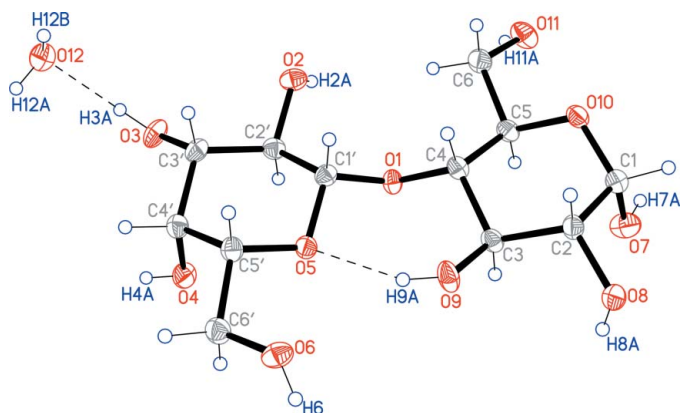
Comment

α-Lactose monohydrate, (I), is the most common form of lactose and may be used as the parent material for at least four different (pseudo)polymorphs of this disaccharide (Garnier *et al.*, 2002; Figura & Epple, 1995). This reducing sugar is built from a moiety of β-D-galactose and a moiety of α-D-glucose, joined by a 1,4 glycosidic bond between C1′ of the galactose and C4 of the glucose unit (Fig. 1).
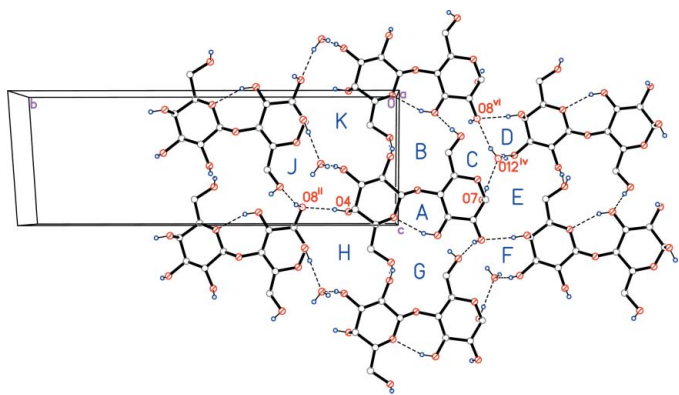

(I)

A search of the Cambridge Structural Database (Allen, 2002; Fletcher *et al.*, 1996; Version 5.26, November 2004 update) highlighted previous research on this hydrate with data collections performed at *ca* 296 K (Beevers & Hansen, 1971; Fries *et al.*, 1971; Noordik *et al.*, 1984). The redetermination of (I) presented here, obtained from low temperature [150 (2) K] single-crystal diffraction data, has resulted in improved precision compared to the previously determined room-temperature structures. Standard uncertainties on C—O and C—C bond lengths are improved to *ca* 0.003 compared to *ca* 0.004 at room temperature, with an improvement to *ca* 0.0017 compared to *ca* 0.002 for standard uncertainties on C–O–C angles. The unit cell volume measured at 150 K [768.85 (14) Å^3] is *ca* 0.88% smaller than that determined at room temperature [775.7 (5) Å^3; Noordik

Figure 1
View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Only hydroxyl H atoms involved in hydrogen bonding are labelled. Dashed lines indicate hydrogen bonds. One intramolecular hydrogen bond is present, *viz.* O9–H9A···O5.
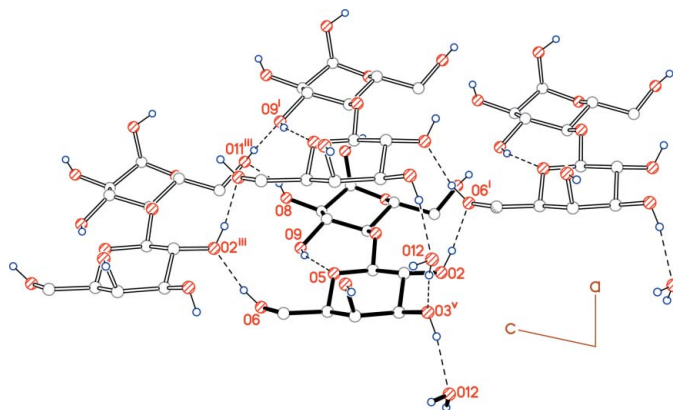

Figure 2
Packing diagram of (I) showing graph-set notation for hydrogen bonding within the crystal structure when viewed along the crystallographic *a* axis. It can be observed that each system of (I), results in motifs: (A) $S(7)$; (B) $R_3^3(16)$; (C) $R_3^3(14)$; (D) $R_3^3(9)$; (E) $R_4^4(18)$; (F) $R_4^3(16)$; (G) $R_3^3(19)$; (H) $R_4^3(18)$; (J) $R_4^4(15)$; (K) $R_5^4(21)$ approximately in the *bc* plane.

Figure 3
View of (I) along the crystallographic *b* axis, showing the remaining hydrogen bonding motifs formed between layers of (I) and water molecules; containing O2—H2A···O6^i, O3—H3A···O12, O6—H6···O2^{iii}, O8—H8A···O11^{iii}, O9—H9A···O5, O11—H11A···O9^i and O12—H12B···O3^v hydrogen bonds [symmetry codes: (i) $x - 1$, y, $z - 1$; (iii) x, y, $1 + z$; (v) $1 + x$, y, z].

et al., 1984], this latter unit-cell volume itself being smaller than that derived from previous measurements.

The unit cell has previously been reported as 7.937 (2) Å, 21.568 (7) Å, 4.815 (1) Å and $\beta = 109.77$ (2)° (Noordik et al., 1984); the unit cell reported here is related to the Noordik unit cell by a simple transformation and is currently regarded as the conventional unit cell, having the shortest possible vectors in the *ac* plane (International Tables for X-ray Crystallography, 1969, Vol. 1).

An examination of the final difference Fourier map reveals a peak of 0.27 e Å^{-3} at a distance of 1.48 Å from C1, close to the equatorial atom H1. Since the α and β anomers establish a 40:60 equilibrium in solution over time, the question arises whether there is a small component of β-lactose present, even though only the α-anomer spontaneously crystallizes below 366.5 K (Walstra & Jenness, 1984). In this present determination, the largest ten difference map features lie in the range 0.22–0.32 e Å^{-3}, so this dubious peak is, in fact, indistinguishable from the noise. This means that, if present at all, the percentage of the β-anomer must be in the low single figures and any significant β component can definitely be ruled out.

Graph-set analysis of the hydrogen-bonding patterns (Bernstein et al., 1995; Etter et al., 1990) within the structure shows the complicated nature of the linking together of the lactose and water molecules. There are 15 different ring motifs involving one molecule of hydrated α-lactose (Figs. 2 and 3). The motifs use either two or three molecules of (I), hydrogen bonding with zero, one or two water molecules. Fig. 4 shows a stacking formation of the lactose molecules when viewed, as a packing plot, along the crystallographic *c* axis. The molecules are held rigidly by a chain, $C_2^2(4)$, of hydrogen bonds between O6–H6A···O2^{iii} and O2–H2A···O6^i [symmetry codes: (i) $x - 1$, y, $z - 1$; (iii) x, y, $1 + z$] propagating along the crystallographic *a* axis and are also linked through hydrogen bonding to water molecules.

As well as those motifs present along the crystallographic *a* axis (Fig. 2), higher order motifs $R_6^6(21)$, $R_4^4(20)$, $R_5^5(20)$, $R_6^6(23)$ and $R_4^4(18)$ can be found between layers of (I) and interconnecting water molecules (Figs. 3 and 4).

Experimental

Colourless X-ray quality crystals of (I) were produced using powdered D-(+)-α-lactose monohydrate (supplied by Fluka Biochemica, Stenheim). A 10% aqueous solution of (I) was prepared as in methods previously studied by Larhrib et al. (2003). This solution was then diluted through addition of acetone, resulting in a 35:65 mixture of 10% lactose solution–acetone. Crystallization occurred upon standing at room temperature over a period of 48 h. A second crystalline sample of (I) was produced by a similar method except that acetone was substituted with a 10% potassium methoxide aqueous solution. Diffraction data from this sample were recorded by the EPSRC National Crystallographic Service, affording very similar unit-cell dimensions.

Crystal data

$C_{12}H_{22}O_{11} \cdot H_2O$
$M_r = 360.31$
Monoclinic, $P2_1$
$a = 4.7830 (5)$ Å
$b = 21.540 (2)$ Å
$c = 7.7599 (8)$ Å
$\beta = 105.911 (2)°$
$V = 768.85 (14)$ Å^3
$Z = 2$

$D_x = 1.556$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3523
  reflections
$\theta = 2.7–28.2°$
$\mu = 0.14$ mm^{-1}
$T = 150 (2)$ K
Block, colourless
$0.53 \times 0.27 \times 0.21$ mm

Data collection

Bruker SMART 1000 CCD
  diffractometer
Narrow-frame ω scans
Absorption correction: multi-scan
  (*SADABS*; Sheldrick, 2003)
  $T_{min} = 0.878$, $T_{max} = 0.971$
6697 measured reflections

1864 independent reflections
1692 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 28.9°$
$h = -6 \rightarrow 6$
$k = -27 \rightarrow 27$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.079$
$S = 1.10$
1864 reflections
231 parameters
H atoms treated by a mixture of
  independent and constrained
  refinement

$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2$
    $+ 0.1957P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1′	1.398 (3)	C1—O7	1.399 (3)
O1—C4	1.437 (3)		
C1′—O1—C4	116.88 (17)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O2—H2A$\cdots$O6^i	0.84	1.85	2.665 (2)	163
O3—H3A$\cdots$O12	0.84	1.89	2.722 (3)	168
O4—H4A$\cdots$O8^{ii}	0.84	1.97	2.806 (3)	173
O6—H6$\cdots$O2^{iii}	0.84	1.90	2.707 (2)	161
O7—H7A$\cdots$O12^{iv}	0.84	1.97	2.772 (2)	161
O8—H8A$\cdots$O11^{iii}	0.84	1.91	2.700 (3)	157
O9—H9A$\cdots$O5	0.84	2.02	2.819 (2)	159
O11—H11A$\cdots$O9^i	0.84	1.92	2.755 (2)	174
O12—H12B$\cdots$O3^v	0.85 (1)	1.89 (2)	2.740 (3)	174 (4)
O12—H12A$\cdots$O8^{vi}	0.84 (1)	2.23 (2)	2.920 (2)	140 (3)

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

Non-water H atoms were placed in geometric positions using a riding model [C—H = 0.99 (methylene H) and 1.00 Å (methine H); O—H = 0.84 Å], and $U_{iso}(H) = 1.2U_{eq}(C)$ and $1.5U_{eq}(O)$. The data set was truncated at $2\theta = 52°$, as only statistically insignificant data were present above this limit. Water H atoms were located in a difference Fourier map and refined using restraints on the O—H bond length
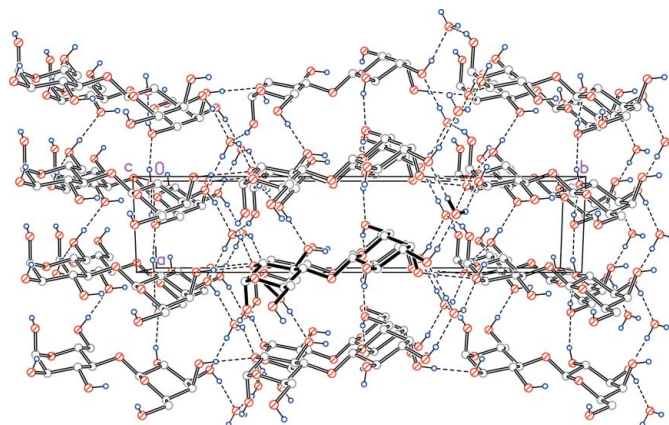

Figure 4
Packing diagram of (I), viewed along the crystallographic *c* axis, showing the hydrogen bonding linking molecules of (I) and water, above and below a central molecule.

[target value 0.840 (15) Å] and the 1,3-distance [target value 1.43 (2) Å] and $U_{iso}(H) = 1.5U_{eq}(O)$. In the absence of significant anomalous dispersion effects, 1526 Friedel pairs were merged during the refinement of (I).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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Monoclinic, $P2_1$
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$\beta = 105.911 (2)°$
$V = 768.85 (14)$ Å^3
$Z = 2$

$F(000) = 384$
$D_x = 1.556$ Mg m^{-3}
Melting point: 220 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3523 reflections
$\theta = 2.7–28.2°$
$\mu = 0.14$ mm^{-1}
$T = 150$ K
Block, colourless
$0.53 \times 0.27 \times 0.21$ mm

Data collection

Bruker SMART 1000 CCD
    diffractometer
Radiation source: sealed tube
Graphite monochromator
ω rotation with narrow frames scans
Absorption correction: multi-scan
    (SADABS; Sheldrick, 2003)
$T_{min} = 0.878$, $T_{max} = 0.971$

6697 measured reflections
1864 independent reflections
1692 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 28.9°$, $\theta_{min} = 1.9°$
$h = -6 \rightarrow 6$
$k = -27 \rightarrow 27$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.079$
$S = 1.10$
1864 reflections
231 parameters
4 restraints
Primary atom site location: structure-invariant
    direct methods

Secondary atom site location: difference Fourier
    map
Hydrogen site location: Geom except H12A&B
    coords freely refined
H atoms treated by a mixture of independent
    and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 0.1957P]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Special details

Experimental. 1526 Friedel pairs. Equivalent reflections and Friedel pairs merged (MERG 4 in *SHELXL*) since Mo radiation & no element heavier than O.

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.0485 (4)	−0.06111 (7)	0.7307 (2)	0.0172 (3)
C1′	0.1611 (5)	−0.01414 (11)	0.7705 (3)	0.0168 (5)
H1′	0.3530	−0.0304	0.7625	0.020*
C2′	0.0574 (5)	0.03808 (11)	0.6356 (3)	0.0182 (5)
H2′	−0.1467	0.0494	0.6322	0.022*
O2	0.0640 (4)	0.01591 (9)	0.4648 (2)	0.0222 (4)
H2A	−0.1065	0.0093	0.4019	0.033*
C3′	0.2515 (5)	0.09504 (11)	0.6874 (3)	0.0189 (5)
H3′	0.4489	0.0855	0.6738	0.023*
O3	0.1249 (4)	0.14411 (8)	0.5683 (2)	0.0242 (4)
H3A	0.2572	0.1660	0.5474	0.036*
C4′	0.2784 (5)	0.11279 (11)	0.8832 (3)	0.0189 (5)
H4′	0.4201	0.1477	0.9200	0.023*
O4	0.0032 (4)	0.13101 (8)	0.9035 (3)	0.0228 (4)
H4A	−0.0208	0.1692	0.8833	0.034*
C5′	0.3853 (5)	0.05660 (12)	1.0023 (3)	0.0179 (5)
H5′	0.5796	0.0435	0.9901	0.022*
C6′	0.4085 (6)	0.06982 (12)	1.1971 (3)	0.0223 (5)
H6′A	0.5526	0.1032	1.2414	0.027*
H6′B	0.2181	0.0841	1.2094	0.027*
O6	0.4960 (4)	0.01480 (9)	1.3010 (2)	0.0237 (4)
H6	0.3859	0.0089	1.3672	0.036*
O5	0.1815 (4)	0.00615 (8)	0.9485 (2)	0.0180 (3)
C1	−0.0864 (5)	−0.25161 (12)	0.8074 (3)	0.0207 (5)
H1	−0.0221	−0.2960	0.8191	0.025*
O7	−0.3771 (4)	−0.24879 (10)	0.8105 (2)	0.0277 (4)
H7A	−0.4841	−0.2626	0.7136	0.042*
C2	0.0961 (5)	−0.21524 (11)	0.9691 (3)	0.0178 (5)
H2	0.3048	−0.2211	0.9721	0.021*
O8	0.0602 (4)	−0.23976 (8)	1.1327 (2)	0.0209 (4)
H8A	−0.0856	−0.2234	1.1546	0.031*
C3	0.0325 (5)	−0.14595 (11)	0.9477 (3)	0.0175 (5)
H3	−0.1683	−0.1379	0.9577	0.021*

O9	0.2382 (4)	−0.11500 (8)	1.0912 (2)	0.0235 (4)
H9A	0.2228	−0.0764	1.0755	0.035*
C4	0.0540 (5)	−0.12383 (11)	0.7645 (3)	0.0168 (5)
H4	0.2609	−0.1262	0.7605	0.020*
C5	−0.1351 (5)	−0.16374 (11)	0.6149 (3)	0.0174 (5)
H5	−0.3432	−0.1599	0.6155	0.021*
C6	−0.1058 (6)	−0.14661 (12)	0.4311 (3)	0.0225 (5)
H6A	−0.1494	−0.1019	0.4084	0.027*
H6B	0.0965	−0.1540	0.4269	0.027*
O11	−0.3010 (4)	−0.18277 (9)	0.2957 (2)	0.0262 (4)
H11A	−0.4400	−0.1604	0.2395	0.039*
O10	−0.0445 (4)	−0.22706 (8)	0.6468 (2)	0.0186 (4)
O12	0.6064 (4)	0.20401 (9)	0.5314 (2)	0.0234 (4)
H12A	0.619 (7)	0.2294 (13)	0.616 (3)	0.035*
H12B	0.764 (5)	0.1835 (14)	0.548 (4)	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0176 (8)	0.0132 (8)	0.0192 (8)	0.0004 (6)	0.0023 (7)	0.0010 (7)
C1′	0.0166 (10)	0.0174 (11)	0.0170 (11)	0.0003 (9)	0.0056 (9)	0.0010 (9)
C2′	0.0184 (11)	0.0182 (11)	0.0183 (12)	0.0015 (9)	0.0054 (9)	0.0003 (9)
O2	0.0227 (9)	0.0293 (10)	0.0142 (8)	0.0004 (8)	0.0041 (7)	0.0012 (7)
C3′	0.0172 (11)	0.0193 (12)	0.0207 (12)	0.0015 (9)	0.0063 (9)	0.0051 (10)
O3	0.0229 (9)	0.0221 (9)	0.0277 (10)	0.0003 (7)	0.0069 (8)	0.0117 (8)
C4′	0.0193 (12)	0.0163 (12)	0.0211 (12)	0.0003 (9)	0.0056 (9)	0.0007 (9)
O4	0.0245 (9)	0.0171 (9)	0.0289 (9)	0.0046 (7)	0.0107 (8)	0.0028 (7)
C5′	0.0161 (11)	0.0179 (11)	0.0184 (11)	−0.0015 (9)	0.0023 (9)	−0.0008 (9)
C6′	0.0260 (12)	0.0208 (12)	0.0189 (12)	−0.0012 (10)	0.0040 (10)	−0.0006 (10)
O6	0.0228 (9)	0.0310 (10)	0.0167 (8)	0.0002 (8)	0.0044 (7)	0.0035 (8)
O5	0.0220 (8)	0.0166 (8)	0.0144 (8)	−0.0029 (7)	0.0032 (6)	−0.0001 (7)
C1	0.0247 (12)	0.0176 (11)	0.0210 (12)	−0.0016 (10)	0.0081 (10)	−0.0024 (10)
O7	0.0239 (9)	0.0367 (11)	0.0227 (9)	−0.0081 (8)	0.0065 (7)	−0.0055 (9)
C2	0.0208 (11)	0.0144 (11)	0.0174 (11)	−0.0002 (9)	0.0039 (9)	0.0004 (9)
O8	0.0260 (9)	0.0173 (9)	0.0200 (9)	0.0021 (7)	0.0070 (7)	0.0022 (7)
C3	0.0201 (11)	0.0157 (11)	0.0145 (11)	0.0006 (8)	0.0012 (9)	−0.0019 (9)
O9	0.0338 (10)	0.0141 (8)	0.0161 (8)	−0.0016 (7)	−0.0041 (7)	−0.0013 (7)
C4	0.0175 (11)	0.0156 (11)	0.0159 (11)	0.0016 (9)	0.0022 (9)	−0.0009 (9)
C5	0.0174 (11)	0.0181 (11)	0.0155 (11)	0.0012 (9)	0.0025 (9)	−0.0022 (9)
C6	0.0290 (13)	0.0220 (13)	0.0151 (12)	−0.0029 (10)	0.0037 (10)	−0.0021 (10)
O11	0.0297 (10)	0.0268 (10)	0.0176 (9)	0.0045 (8)	−0.0013 (7)	−0.0038 (8)
O10	0.0217 (8)	0.0167 (8)	0.0176 (8)	0.0004 (6)	0.0055 (7)	−0.0032 (6)
O12	0.0228 (9)	0.0235 (9)	0.0225 (9)	0.0004 (7)	0.0039 (8)	0.0044 (7)

Geometric parameters (Å, º)

O1—C1′	1.398 (3)	C1—O7	1.399 (3)
O1—C4	1.437 (3)	C1—O10	1.418 (3)

C1′—O5	1.427 (3)	C1—C2	1.533 (3)
C1′—C2′	1.524 (3)	C1—H1	1.0000
C1′—H1′	1.0000	O7—H7A	0.8400
C2′—O2	1.417 (3)	C2—O8	1.428 (3)
C2′—C3′	1.524 (3)	C2—C3	1.523 (3)
C2′—H2′	1.0000	C2—H2	1.0000
O2—H2A	0.8400	O8—H8A	0.8400
C3′—O3	1.425 (3)	C3—O9	1.432 (3)
C3′—C4′	1.538 (3)	C3—C4	1.529 (3)
C3′—H3′	1.0000	C3—H3	1.0000
O3—H3A	0.8400	O9—H9A	0.8400
C4′—O4	1.423 (3)	C4—C5	1.526 (3)
C4′—C5′	1.524 (3)	C4—H4	1.0000
C4′—H4′	1.0000	C5—O10	1.432 (3)
O4—H4A	0.8400	C5—C6	1.516 (3)
C5′—O5	1.443 (3)	C5—H5	1.0000
C5′—C6′	1.512 (3)	C6—O11	1.430 (3)
C5′—H5′	1.0000	C6—H6A	0.9900
C6′—O6	1.430 (3)	C6—H6B	0.9900
C6′—H6′A	0.9900	O11—H11A	0.8400
C6′—H6′B	0.9900	O12—H12A	0.842 (14)
O6—H6	0.8400	O12—H12B	0.851 (14)
C1′—O1—C4	116.88 (17)	O7—C1—O10	112.3 (2)
O1—C1′—O5	106.85 (17)	O7—C1—C2	108.01 (19)
O1—C1′—C2′	107.69 (18)	O10—C1—C2	110.05 (19)
O5—C1′—C2′	111.22 (19)	O7—C1—H1	108.8
O1—C1′—H1′	110.3	O10—C1—H1	108.8
O5—C1′—H1′	110.3	C2—C1—H1	108.8
C2′—C1′—H1′	110.3	C1—O7—H7A	109.5
O2—C2′—C1′	107.89 (19)	O8—C2—C3	113.0 (2)
O2—C2′—C3′	110.5 (2)	O8—C2—C1	111.00 (19)
C1′—C2′—C3′	110.67 (19)	C3—C2—C1	111.1 (2)
O2—C2′—H2′	109.3	O8—C2—H2	107.1
C1′—C2′—H2′	109.3	C3—C2—H2	107.1
C3′—C2′—H2′	109.3	C1—C2—H2	107.1
C2′—O2—H2A	109.5	C2—O8—H8A	109.5
O3—C3′—C2′	107.83 (19)	O9—C3—C2	107.30 (19)
O3—C3′—C4′	111.2 (2)	O9—C3—C4	111.76 (19)
C2′—C3′—C4′	109.9 (2)	C2—C3—C4	110.1 (2)
O3—C3′—H3′	109.3	O9—C3—H3	109.2
C2′—C3′—H3′	109.3	C2—C3—H3	109.2
C4′—C3′—H3′	109.3	C4—C3—H3	109.2
C3′—O3—H3A	109.5	C3—O9—H9A	109.5
O4—C4′—C5′	108.6 (2)	O1—C4—C5	106.83 (18)
O4—C4′—C3′	110.4 (2)	O1—C4—C3	110.87 (19)
C5′—C4′—C3′	108.96 (19)	C5—C4—C3	110.79 (19)
O4—C4′—H4′	109.6	O1—C4—H4	109.4

C5′—C4′—H4′	109.6	C5—C4—H4	109.4
C3′—C4′—H4′	109.6	C3—C4—H4	109.4
C4′—O4—H4A	109.5	O10—C5—C6	107.02 (19)
O5—C5′—C6′	106.82 (19)	O10—C5—C4	108.48 (18)
O5—C5′—C4′	109.58 (18)	C6—C5—C4	113.0 (2)
C6′—C5′—C4′	112.2 (2)	O10—C5—H5	109.4
O5—C5′—H5′	109.4	C6—C5—H5	109.4
C6′—C5′—H5′	109.4	C4—C5—H5	109.4
C4′—C5′—H5′	109.4	O11—C6—C5	110.4 (2)
O6—C6′—C5′	109.7 (2)	O11—C6—H6A	109.6
O6—C6′—H6′A	109.7	C5—C6—H6A	109.6
C5′—C6′—H6′A	109.7	O11—C6—H6B	109.6
O6—C6′—H6′B	109.7	C5—C6—H6B	109.6
C5′—C6′—H6′B	109.7	H6A—C6—H6B	108.1
H6′A—C6′—H6′B	108.2	C6—O11—H11A	109.5
C6′—O6—H6	109.5	C1—O10—C5	113.47 (18)
C1′—O5—C5′	111.85 (17)	H12A—O12—H12B	110 (2)
C4—O1—C1′—O5	−93.4 (2)	O7—C1—C2—O8	57.8 (3)
C4—O1—C1′—C2′	147.00 (19)	O10—C1—C2—O8	−179.21 (18)
O1—C1′—C2′—O2	−67.6 (2)	O7—C1—C2—C3	−68.7 (3)
O5—C1′—C2′—O2	175.61 (17)	O10—C1—C2—C3	54.2 (3)
O1—C1′—C2′—C3′	171.40 (18)	O8—C2—C3—O9	62.2 (3)
O5—C1′—C2′—C3′	54.6 (3)	C1—C2—C3—O9	−172.33 (19)
O2—C2′—C3′—O3	67.0 (2)	O8—C2—C3—C4	−175.98 (19)
C1′—C2′—C3′—O3	−173.61 (19)	C1—C2—C3—C4	−50.5 (2)
O2—C2′—C3′—C4′	−171.67 (19)	C1′—O1—C4—C5	−143.26 (19)
C1′—C2′—C3′—C4′	−52.2 (3)	C1′—O1—C4—C3	95.9 (2)
O3—C3′—C4′—O4	55.1 (3)	O9—C3—C4—O1	−69.7 (2)
C2′—C3′—C4′—O4	−64.3 (2)	C2—C3—C4—O1	171.10 (19)
O3—C3′—C4′—C5′	174.24 (19)	O9—C3—C4—C5	171.83 (19)
C2′—C3′—C4′—C5′	54.9 (2)	C2—C3—C4—C5	52.7 (2)
O4—C4′—C5′—O5	60.5 (2)	O1—C4—C5—O10	−178.42 (18)
C3′—C4′—C5′—O5	−59.8 (2)	C3—C4—C5—O10	−57.6 (2)
O4—C4′—C5′—C6′	−57.9 (3)	O1—C4—C5—C6	63.1 (2)
C3′—C4′—C5′—C6′	−178.2 (2)	C3—C4—C5—C6	−176.0 (2)
O5—C5′—C6′—O6	57.2 (2)	O10—C5—C6—O11	64.3 (2)
C4′—C5′—C6′—O6	177.2 (2)	C4—C5—C6—O11	−176.3 (2)
O1—C1′—O5—C5′	−177.84 (17)	O7—C1—O10—C5	58.4 (3)
C2′—C1′—O5—C5′	−60.6 (2)	C2—C1—O10—C5	−62.0 (2)
C6′—C5′—O5—C1′	−174.86 (18)	C6—C5—O10—C1	−174.31 (18)
C4′—C5′—O5—C1′	63.4 (2)	C4—C5—O10—C1	63.5 (2)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O6[i]	0.84	1.85	2.665 (2)	163
O3—H3A···O12	0.84	1.89	2.722 (3)	168

O4—H4*A*···O8ⁱⁱ	0.84	1.97	2.806 (3)	173
O6—H6···O2ⁱⁱⁱ	0.84	1.90	2.707 (2)	161
O7—H7*A*···O12ⁱᵛ	0.84	1.97	2.772 (2)	161
O8—H8*A*···O11ⁱⁱⁱ	0.84	1.91	2.700 (3)	157
O9—H9*A*···O5	0.84	2.02	2.819 (2)	159
O11—H11*A*···O9ⁱ	0.84	1.92	2.755 (2)	174
O12—H12*B*···O3ᵛ	0.85 (1)	1.89 (2)	2.740 (3)	174 (4)
O12—H12*A*···O8ᵛⁱ	0.84 (1)	2.23 (2)	2.920 (2)	140 (3)

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