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3,3′:5,6-Di-*O*-isopropylidene-3-*C*-hydroxymethyl-D-*allono*-1,4-lactone: an organic structure containing large unoccupied voids

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.046
wR factor = 0.044
Data-to-parameter ratio = 10.0

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

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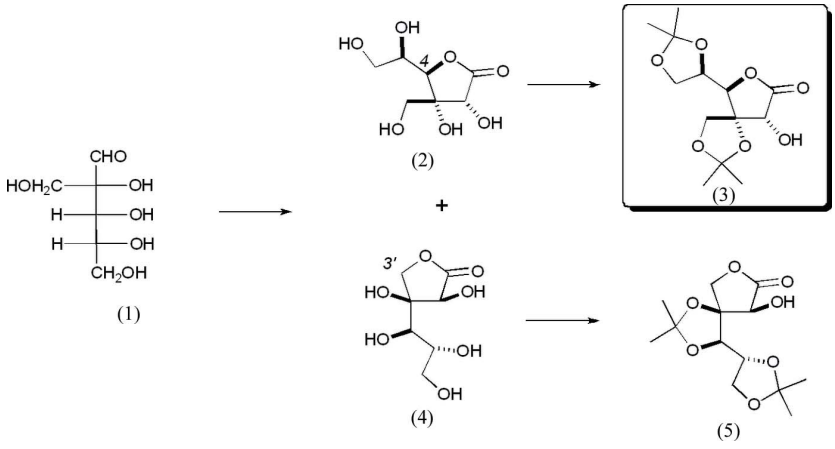
The Kiliani reaction of D-hamamelose with sodium cyanide, followed by acetonation, affords crystalline 3,3′:5,6-di-*O*-isopropylidene-3-*C*-hydroxymethyl-D-allono-1,4-lactone, $C_{13}H_{20}O_7$, a carbon-branched sugar with potential as an enantiomerically pure carbohydrate scaffold. The lactone has one single free hydroxyl group unprotected, with six other functional groups protected in a single step as ketals or esters. The resulting crystal structure is unusual in that it contains large voids (544 Å^3) within which there is no evidence of included solvent.

Comment

At present, there are very few accessible branched carbohydrate scaffolds (Lichtenthaler & Peters, 2004; Bols, 1996) for use in the synthesis of complex enantiomerically pure targets (Simone *et al.*, 2005). The reactions of calcium oxide on Amadori 1-deoxyamino-ketoses (Hotchkiss *et al.*, 2006) and the Kiliani reaction of cyanide with ketoses (Hotchkiss *et al.*, 2004; Soengas *et al.*, 2005) allow the preparation of 2-*C*-methyl and 2-*C*-hydroxymethyl lactones in relatively short sequences. However, syntheses of carbohydrates bearing a carbon branch at C-3 are very rare (Bream *et al.*, 2006). One approach to such chirons is the Kiliani cyanide reaction on 2-*C*-hydroxymethyl sugars [such as hamamelose (1)] to produce 3-*C*-hydroxymethyl lactones [such as (2) and (4)]. The experimental details for the Kiliani reaction of D-hamamelose (1) with cyanide to give a mixture of the two branched sugar lactones (2) and (4), followed by treatment with dimethoxypropane to afford a separable mixture of the two diacetonides (3) and (5), have been reported in a previous paper (Parker *et al.*, 2006).



A significant number of ambiguities arise from the formation of possible lactones, the sites for the formation of cyclic

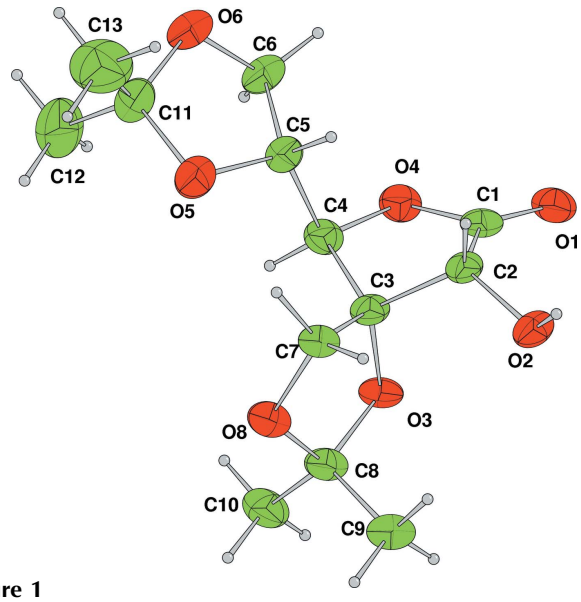


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

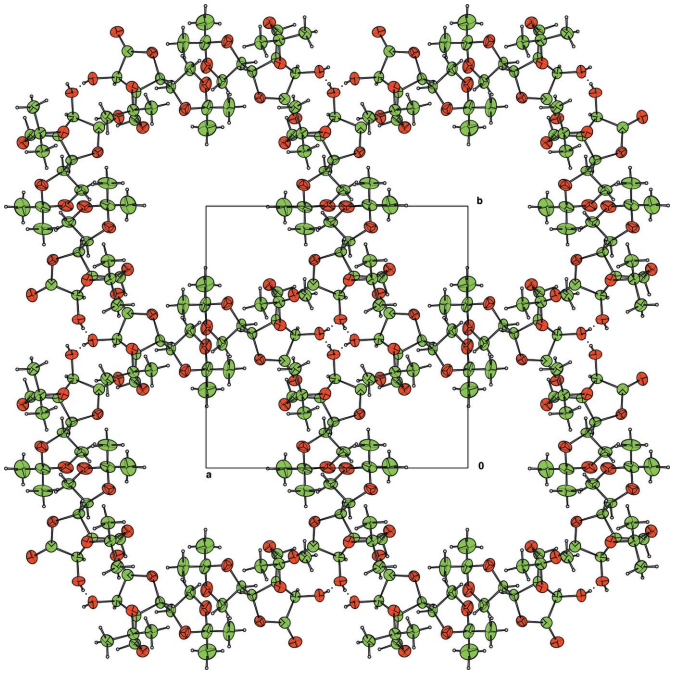


Figure 2
Packing diagam of the title compound viewed along *c*. The residual electron density in the void has a maximum value of 0.22 e Å^{-3}. Hydrogen bonds are drawn as dotted lines

ketals and the sizes of both the lactone and ketal rings. This is an area in which X-ray crystallography is needed to have any confidence at all in the structures of diacetonides obtained by this short procedure. The crystal structure of the altrono-diacetonide (5), formed from the lactone (4) derived by cyclization of the branched C-3′ hydroxymethyl group on to the carboxylic acid, has been established by X-ray crystallography (Parker *et al.*, 2006). This paper firmly assigns the structure of the second crystalline product as the branched

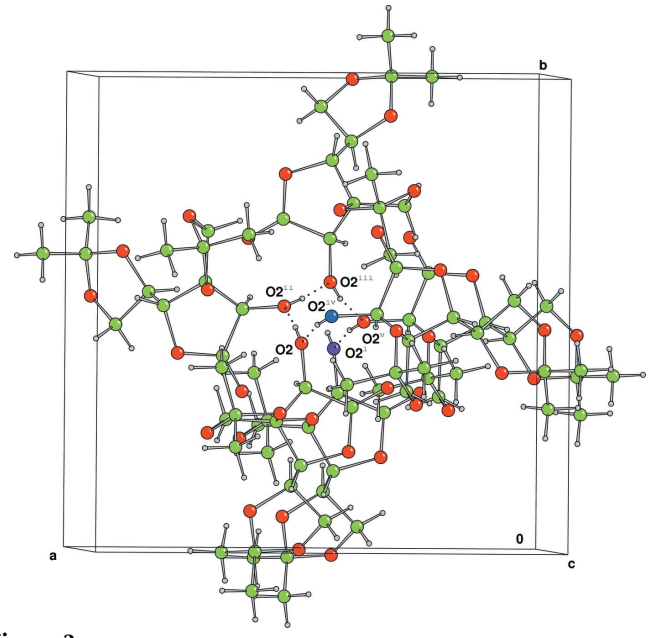


Figure 3
Oblique packing diagram of the title compound, showing the hydrogen-bonded helix which is the main structural feature. The ends of one turn of the helix are coloured blue and purple. Operators involved in forming the helix are: (i) $x, y, z+1$; (ii) $-y+1, x, z+\frac{1}{4}$; (iii) $-x+1, -y+1, z+\frac{1}{2}$; (iv) $y, -x+1, z-\frac{1}{4}$; (v) $y, -x+1, z+\frac{3}{4}$. Hydrogen bonds are drawn as dotted lines.

allono-lactone (3) formed from lactone formation from the C-4 hydroxyl group; the absolute configuration of (3) is determined by the use of D-ribose as the starting material for the synthesis. It is noteworthy that both lactones (3) and (5) have only the C-2 hydroxyl group unprotected; the sequence provides access to two sugars with seven functional groups but with six of the them protected in one simple step.

The component molecules have no unusual torsion angles, and show no evidence of internal strain. The relatively large anisotropic displacement parameters for the methyl groups and O atoms in the acetonide protecting group may indicate some ring fluxion (Fig. 1).

The crystal structure is unusual in that it contains substantial voids (544 Å^3) within which there is no evidence for included solvent (Fig. 2). The voids are big enough to have contained dichloromethane, but the maximum residual electron density is only 0.2 e Å^{-3}. We do not know if the voids in the dry crystals ever contained solvent, though generally solvent loss from organic crystals is associated with either a total loss of crystallinity, or at least a degradation of the crystal quality. In this case the crystals remained glass-clear.

The structure consists of a tight helix (Fig. 3), involving O—H$\cdots$O^{i} hydrogen bonds (Table 1), which runs parallel to the *c* axis at ($\frac{1}{2}$, $\frac{1}{2}$, *z*). The main parts of the molecule hang off this backbone like leaves from a tree. The tips of the 'leaves' of four separate helices meet to form a second helix at (0, 0, *z*). The 'leaves' of each pair of adjacent hydrogen-bonded helices (separated by $\frac{z}{2}$ because of the 4_1 axis) interleave along (0, 0, *z*), but there is no evidence for particularly strong interactions at these points.

Experimental

The title compound, (3), was crystallized by dissolving it in dichloromethane, adding a few drops of cyclohexane and allowing the slow competitive evaporation of the two solvents until needle-like colourless crystals formed [m.p. 353 K (dichloromethane/cyclohexane)]. MS–ES$^-$ (m/z): 287.2 ([M − H]$^-$, 15%); HRMS (MS ES$^+$): found 311.1101 [M + Na]$^+$ $C_{13}H_{20}NaO_7$ requires 311.1101; $[\alpha]_D^{23}$: +5.5 (c 1.25 in chloroform); ν_{max} (thin film): 3445 (*br*, OH), 2989 (CH_2, CH_3), 1800 (C═O) cm^{-1}; δ_H (C_6D_6, 400 MHz): 1.20, 1.31, 1.36, 1.38 [12H, 4 × *s*, 2 × C(CH_3)$_2$], 3.30–3.40 (1H, *br s*, OH-2), 3.51 (1H, *ddd*, $J_{H\text{-}5,H\text{-}4}$ = 8.5 Hz, $J_{H\text{-}5,H\text{-}6}$ = 6.7 Hz, $J_{H\text{-}5,H\text{-}6'}$ = 5.3 Hz, H-5), 3.75 (1H, *dd*, $J_{H\text{-}6,H\text{-}6'}$ = 9.5 Hz, $J_{H\text{-}6,H\text{-}5}$ = 6.6 Hz, H-6), 3.82 (1H, *dd*, $J_{H\text{-}6',H\text{-}6}$ = 9.5 Hz, $J_{H\text{-}6',H\text{-}5}$ = 5.3 Hz, H-6′), 3.97 (1H, *d*, $J_{H\text{-}3,H\text{-}3'}$ = 10.0 Hz H-3), 4.20 (1H, *s*, H-2), 4.26 (1H, *d*, $J_{H\text{-}4,H\text{-}5}$ = 8.6 Hz, H-4), 4.55 (1H, *d*, $J_{H\text{-}3',H\text{-}3}$ = 10.0 Hz, H-3′); δ_C (C_6D_6, 100 MHz): 24.8, 25.5, 26.5, 26.9 [2 × C(CH_3)$_2$], 65.3 (C-3′), 65.8 (C-6), 67.0 (C-2), 73.7 (C-5), 84.9 (C-3), 85.0 (C-4), 110.7, 111.2 [2 × C(CH_3)$_2$], 173.2 (C═O).

Crystal data

$C_{13}H_{20}O_7$
M_r = 288.30
Tetragonal, $P4_1$
a = 14.1641 (4) Å
c = 9.2045 (3) Å
V = 1846.62 (10) Å^3
Z = 4
D_x = 1.037 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 150 K
Needle, colourless
0.44 × 0.12 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.96, T_{max} = 0.99
21907 measured reflections
2229 independent reflections
1802 reflections with $I > 3\sigma(I)$
R_{int} = 0.068
θ_{max} = 27.6°

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.044
S = 1.04
1802 reflections
181 parameters
H-atom parameters constrained
$w = [1 - (F_o - F_c)^2/36\sigma^2(F)]^2/[1.09T_0(x) + 0.255T_1(x) + 0.726T_2(x)]$ where T_i are Chebychev polynomials and $x = F_c/F_{max}$ (Prince, 1982; Watkin, 1994) Modified Chebychev polynomial (Watkin, 1994; Prince, 1982)
$(\Delta/\sigma)_{max}$ = 0.010
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1···O2^{i}	0.80	1.91	2.679 (2)	160

Symmetry code: (i) $-y+1, x, z+\frac{1}{4}$.

In the absence of significant anomalous scattering, Friedel pairs were merged and the absolute configuration assigned from the starting materials The H atoms were all located in a difference map, but those attached to C 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 = 0.93–0.98, O—H = 0.82 Å and U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom)], after which the positions were refined with riding constraints.

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*.

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References

Altomare, A., Cascarano, G., Giacovazzo, G., 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.
Bols, M. (1996). *Carbohydrate Building Blocks*. New York: John Wiley & Sons.
Bream, R., Watkin, D., Soengas, R., Eastwick-Field, V. & Fleet, G. W. J. (2006). *Acta Cryst.* E**62**, o977–o979.
Hotchkiss, D. J., Jenkinson, S. F., Storer, R., Heinz, T. & Fleet, G. W. J. (2006). *Tetrahedron Lett.* **47**, 315–318.
Hotchkiss, D. J., Soengas, R., Simone, M. I., van Ameijde, J., Hunter, S., Cowley, A. R. & Fleet, G. W. J. (2004). *Tetrahedron Lett.* **45**, 9461–9464.
Lichtenthaler, F. W. & Peters, S. (2004). *C. R. Chim.* **7**, 65–90.
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 and R. M. Sweet, pp. 307–326. New York: Academic Press.
Parker, S. G., Watkin, D. J., Simone, M. I. & Fleet, G. W. J. (2006). *Acta Cryst.* E**62**, o3961–o3963.
Prince, E. (1982). *Mathematical Techniques in Crystallography and Materials Science*. New York: Springer-Verlag.
Simone, M. I., Soengas, R., Newton, C. R., Watkin, D. J. & Fleet, G. W. J. (2005). *Tetrahedron Lett.* **46**, 5761–5765.
Soengas, R., Izumori, K., Simone, M. I., Watkin, D. J., Skytte, U. P., Soetaert, W. & Fleet, G. W. J. (2005). *Tetrahedron Lett.* **46**, 5755–5759.
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.

supporting information

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Hall symbol: P 4w
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$V = 1846.62\ (10)$ Å^3
$Z = 4$
$F(000) = 616$
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Cell parameters from 21907 reflections
$\theta = 5$–$28°$
$\mu = 0.08$ mm^{-1}
$T = 150$ K
Needle, colourless
0.44 × 0.12 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Graphite monochromator
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1802 reflections with $I > 3\sigma(I)$
$R_{int} = 0.068$
$\theta_{max} = 27.6°$, $\theta_{min} = 5.2°$
$h = -12 \rightarrow 13$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 11$

Refinement

Refinement on F
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.044$
$S = 1.04$
1802 reflections
181 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
Modified Chebychev polynomial (Watkin, 1994; *P*rince, 1982) *w*ith coefficients 1.09 0.255 0.726 and Robust Weighting (*P*rince, 1982); W = [*w*eight][1-(δF/6σF)^2]^2
$(\Delta/\sigma)_{max} = 0.010$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.40908 (17)	0.29983 (17)	0.2555 (3)	0.0322
C2	0.50778 (16)	0.34055 (15)	0.2595 (2)	0.0292
C3	0.56325 (17)	0.26748 (16)	0.1741 (2)	0.0306

C4	0.51393 (17)	0.17588 (17)	0.2218 (3)	0.0340
C5	0.54440 (19)	0.13445 (17)	0.3650 (3)	0.0384
C6	0.4755 (2)	0.06101 (19)	0.4243 (4)	0.0493
O1	0.33445 (12)	0.34056 (13)	0.2657 (2)	0.0401
O2	0.51303 (12)	0.43275 (11)	0.20539 (19)	0.0336
O3	0.54536 (12)	0.28276 (12)	0.02234 (18)	0.0340
O4	0.41413 (12)	0.20543 (12)	0.2386 (2)	0.0367
O5	0.62892 (15)	0.08096 (13)	0.3439 (3)	0.0522
O6	0.53620 (16)	0.00210 (14)	0.5075 (3)	0.0579
C7	0.67037 (18)	0.27177 (18)	0.1852 (3)	0.0356
O8	0.69994 (13)	0.24162 (13)	0.0446 (2)	0.0409
C8	0.63374 (18)	0.27831 (18)	−0.0555 (3)	0.0356
C9	0.6603 (2)	0.3780 (2)	−0.1031 (3)	0.0434
C10	0.6248 (2)	0.2097 (2)	−0.1803 (3)	0.0477
C11	0.6237 (2)	−0.0037 (2)	0.4304 (3)	0.0510
C12	0.6225 (4)	−0.0873 (2)	0.3307 (5)	0.0805
C13	0.7021 (3)	−0.0043 (3)	0.5393 (4)	0.0793
H1	0.5317	0.4678	0.2677	0.0507*
H21	0.5308	0.3403	0.3595	0.0347*
H41	0.5201	0.1275	0.1480	0.0411*
H51	0.5523	0.1854	0.4369	0.0454*
H61	0.4264	0.0889	0.4849	0.0586*
H62	0.4459	0.0254	0.3444	0.0580*
H71	0.6906	0.3361	0.2042	0.0413*
H72	0.6947	0.2293	0.2595	0.0417*
H91	0.7203	0.3782	−0.1548	0.0642*
H92	0.6673	0.4189	−0.0191	0.0647*
H93	0.6102	0.4041	−0.1660	0.0643*
H101	0.6850	0.2058	−0.2287	0.0703*
H102	0.5776	0.2310	−0.2491	0.0705*
H103	0.6073	0.1473	−0.1420	0.0706*
H121	0.6216	−0.1447	0.3883	0.1191*
H122	0.6795	−0.0864	0.2731	0.1185*
H123	0.5676	−0.0845	0.2679	0.1184*
H131	0.6983	−0.0602	0.6003	0.1178*
H132	0.7623	−0.0033	0.4884	0.1177*
H133	0.6982	0.0510	0.5999	0.1178*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0403 (13)	0.0401 (12)	0.0161 (8)	−0.0017 (10)	0.0017 (9)	0.0035 (9)
C2	0.0405 (12)	0.0301 (11)	0.0169 (9)	0.0024 (9)	−0.0033 (9)	0.0022 (9)
C3	0.0429 (13)	0.0302 (11)	0.0188 (10)	−0.0015 (9)	−0.0003 (9)	0.0012 (8)
C4	0.0432 (13)	0.0297 (11)	0.0292 (11)	−0.004 (1)	0.0058 (9)	−0.0041 (9)
C5	0.0508 (14)	0.0277 (11)	0.0368 (12)	0.0027 (10)	0.0091 (11)	0.0047 (10)
C6	0.0598 (17)	0.0355 (13)	0.0526 (17)	0.0019 (12)	0.0124 (14)	0.0173 (13)
O1	0.0380 (9)	0.0546 (10)	0.0278 (8)	0.0050 (8)	0.0042 (7)	0.0055 (8)

O2	0.0501 (10)	0.0280 (8)	0.0228 (7)	−0.0023 (7)	−0.0088 (7)	0.0007 (6)
O3	0.0403 (9)	0.0442 (9)	0.0174 (7)	−0.0029 (7)	0.0011 (6)	0.0016 (7)
O4	0.0390 (9)	0.0369 (9)	0.0343 (9)	−0.0044 (7)	0.0035 (7)	0.0001 (7)
O5	0.0550 (12)	0.0410 (10)	0.0606 (13)	0.0091 (9)	0.0166 (10)	0.0154 (9)
O6	0.0734 (13)	0.0437 (10)	0.0566 (13)	0.0090 (10)	0.0162 (12)	0.0237 (10)
C7	0.0403 (13)	0.0397 (13)	0.0269 (11)	0.0011 (10)	0.0059 (10)	0.0042 (10)
O8	0.0432 (10)	0.0476 (10)	0.0319 (9)	0.0031 (8)	0.0084 (7)	0.0024 (8)
C8	0.0410 (13)	0.0407 (13)	0.0250 (11)	−0.0021 (10)	0.0054 (10)	0.0018 (10)
C9	0.0471 (15)	0.0496 (15)	0.0335 (12)	−0.0063 (12)	0.0070 (11)	0.0057 (12)
C10	0.0611 (17)	0.0499 (15)	0.0320 (13)	0.0003 (13)	0.0107 (12)	−0.0074 (12)
C11	0.0686 (19)	0.0385 (14)	0.0458 (15)	0.0130 (13)	0.0028 (14)	0.0082 (12)
C12	0.120 (3)	0.0417 (17)	0.080 (3)	0.0111 (19)	0.011 (2)	−0.0076 (18)
C13	0.078 (2)	0.103 (3)	0.057 (2)	0.011 (2)	−0.0075 (19)	0.011 (2)

Geometric parameters (Å, º)

C1—C2	1.513 (3)	O6—C11	1.430 (4)
C1—O1	1.208 (3)	C7—O8	1.426 (3)
C1—O4	1.348 (3)	C7—H71	0.972
C2—C3	1.519 (3)	C7—H72	0.974
C2—O2	1.400 (3)	O8—C8	1.413 (3)
C2—H21	0.976	C8—C9	1.526 (4)
C3—C4	1.538 (3)	C8—C10	1.510 (4)
C3—O3	1.436 (2)	C9—H91	0.974
C3—C7	1.522 (4)	C9—H92	0.971
C4—C5	1.506 (4)	C9—H93	0.987
C4—O4	1.482 (3)	C10—H101	0.963
C4—H41	0.968	C10—H102	0.970
C5—C6	1.527 (4)	C10—H103	0.984
C5—O5	1.430 (3)	C11—C12	1.499 (5)
C5—H51	0.986	C11—C13	1.497 (5)
C6—O6	1.422 (4)	C12—H121	0.972
C6—H61	0.975	C12—H122	0.965
C6—H62	0.985	C12—H123	0.970
O2—H1	0.803	C13—H131	0.972
O3—C8	1.444 (3)	C13—H132	0.972
O5—C11	1.441 (3)	C13—H133	0.964
C2—C1—O1	128.7 (2)	C3—C7—H72	112.0
C2—C1—O4	109.39 (19)	O8—C7—H72	110.4
O1—C1—O4	121.9 (2)	H71—C7—H72	110.5
C1—C2—C3	101.88 (18)	C7—O8—C8	106.66 (18)
C1—C2—O2	113.35 (18)	O3—C8—O8	105.54 (17)
C3—C2—O2	115.11 (18)	O3—C8—C9	108.4 (2)
C1—C2—H21	109.2	O8—C8—C9	111.4 (2)
C3—C2—H21	108.2	O3—C8—C10	109.5 (2)
O2—C2—H21	108.8	O8—C8—C10	108.3 (2)
C2—C3—C4	101.08 (18)	C9—C8—C10	113.4 (2)

C2—C3—O3	108.02 (18)	C8—C9—H91	110.9
C4—C3—O3	108.96 (19)	C8—C9—H92	110.4
C2—C3—C7	117.0 (2)	H91—C9—H92	107.3
C4—C3—C7	117.86 (19)	C8—C9—H93	109.7
O3—C3—C7	103.63 (18)	H91—C9—H93	109.9
C3—C4—C5	116.7 (2)	H92—C9—H93	108.6
C3—C4—O4	103.00 (18)	C8—C10—H101	108.4
C5—C4—O4	106.95 (19)	C8—C10—H102	110.7
C3—C4—H41	110.8	H101—C10—H102	109.1
C5—C4—H41	108.2	C8—C10—H103	109.1
O4—C4—H41	111.1	H101—C10—H103	109.7
C4—C5—C6	113.3 (2)	H102—C10—H103	109.8
C4—C5—O5	109.1 (2)	O5—C11—O6	105.7 (2)
C6—C5—O5	102.86 (19)	O5—C11—C12	108.6 (3)
C4—C5—H51	109.5	O6—C11—C12	109.9 (3)
C6—C5—H51	109.4	O5—C11—C13	109.7 (3)
O5—C5—H51	112.6	O6—C11—C13	108.2 (3)
C5—C6—O6	101.9 (2)	C12—C11—C13	114.4 (3)
C5—C6—H61	112.6	C11—C12—H121	109.1
O6—C6—H61	111.2	C11—C12—H122	108.5
C5—C6—H62	110.7	H121—C12—H122	108.8
O6—C6—H62	111.0	C11—C12—H123	110.0
H61—C6—H62	109.3	H121—C12—H123	110.4
C2—O2—H1	109.9	H122—C12—H123	110.1
C3—O3—C8	108.84 (18)	C11—C13—H131	110.5
C4—O4—C1	110.04 (17)	C11—C13—H132	109.1
C5—O5—C11	108.8 (2)	H131—C13—H132	109.8
C6—O6—C11	106.9 (2)	C11—C13—H133	109.8
C3—C7—O8	102.68 (19)	H131—C13—H133	109.0
C3—C7—H71	110.1	H132—C13—H133	108.5
O8—C7—H71	110.9		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1···O2^{i}	0.80	1.91	2.679 (2)	160

Symmetry code: (i) −*y*+1, *x*, *z*+1/4.