IUCrData

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(2*S*,3*S*,4*R*,4a′*R*,5*R*,5a′*R*,11a′*R*,12′*S*,12a′*R*)-5-(Acetoxymethyl)-2′,2′,10′,10′-tetramethyloctahydro-3*H*,8′*H*-spiro[furan-2,7′-[1,3]dioxino[4′,5′:5,6]pyrano[3,2-*d*][1,3,6]trioxocine]-3,4,12′-triyl triacetate

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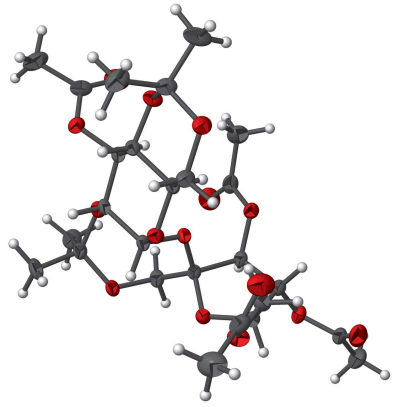
Keywords: crystal structure; sucrose derivative; absolute configuration; C—H···O hydrogen bonding.

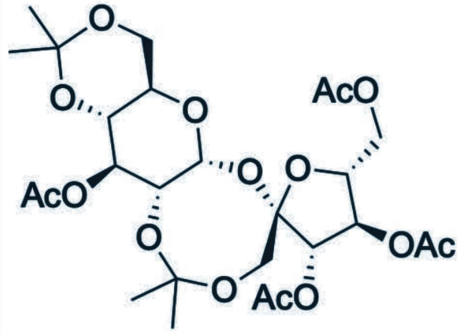
Structural data: full structural data are available from iucrdata.iucr.org

While the crystal structure analysis of the title compound, $C_{26}H_{38}O_{15}$, a synthetic derivative of sucrose, was originally reported 40 years ago [Drew *et al.* (1979). *Carbohydr. Res.* **71**, 35–42], the present work has allowed for the determination of its absolute configuration through the application of resonant scattering techniques.

3D view



Chemical scheme



Structure description

Sucrose and certain derivatives are profoundly important commodity chemicals in, for example, the food, nutraceutical, cosmetic, dental and pharmaceutical industries (Farrán *et al.*, 2015). However, selective manipulation of the eight distinct hydroxyl groups within the parent compound is challenging (Queneau *et al.*, 2008). Accordingly, we were attracted to the title derivative, a previously reported compound (Fanton *et al.*, 1981; Khan & Mufti, 1975; Poschalko *et al.*, 2003), as a readily accessible one that could serve as the starting point for selective re-functionalization of the sucrose framework and so affording a range of single-compound derivatives. To such ends we required detailed structural information on the title compound, including its solid-state properties [so as to inform proposed mechanochemical studies (Achar *et al.*, 2017)] and thus undertook the high-resolution single-crystal X-ray analysis reported here (Fig. 1). The compound crystallized in the chiral monoclinic space group $P2_1$. The absolute configuration was determined by resonant scattering. Upon refinement of the Flack parameter, this calculated to the unambiguous value of 0.05 (8).

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

D—H···A	D—H	H···A	D···A	D—H···A
C1′—H1′A···O17	0.97	2.40	3.154 (3)	134
C6′—H6′B···O1	0.97	2.57	3.189 (3)	122
C6—H6B···O23	0.97	2.41	3.318 (3)	156
C12—H12A···O20^{i}	0.96	2.58	3.432 (4)	148
C21—H21A···O11^{ii}	0.96	2.59	3.433 (4)	146
C24—H24C···O23^{iii}	0.96	2.49	3.339 (4)	147

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

A single-crystal, room-temperature X-ray analysis of the title compound was reported nearly forty years ago (Drew *et al.*, 1979) but its absolute configuration was not determined by that means. For the purposes of the present study, a sample of this sucrose derivative was prepared by the same means as used earlier (Drew *et al.*, 1979), involving initial diacetonide formation followed by acetylation of the four remaining free hydroxyl groups (Khan & Mufti, 1975). Crystals suitable for analysis were grown, using vapour-diffusion techniques, from diethyl ether/40–60 petroleum spirits and the derived spectroscopic data matched those reported previously. The *R* factor arising from the present study at 150 K was superior to that obtained earlier (3.66% *versus* 5.5%) and this is mirrored through the more accurate unit-cell parameters. As noted in the earlier study (Drew *et al.*, 1979), the eight-membered and trioxygenated ring embedded within this sucrose derivative is an unusual structural feature, as is the conformation of the tetrahydrofuranyl residue wherein the ring oxygen atom (O2′) is *exo*-related to C6′. There are a number of intramolecular C—H···O contacts present (Table 1).

In the crystal, molecules are linked by a number of C—H···O hydrogen bonds, forming slabs parallel to the *ab* plane (Table 1 and Fig. 2).

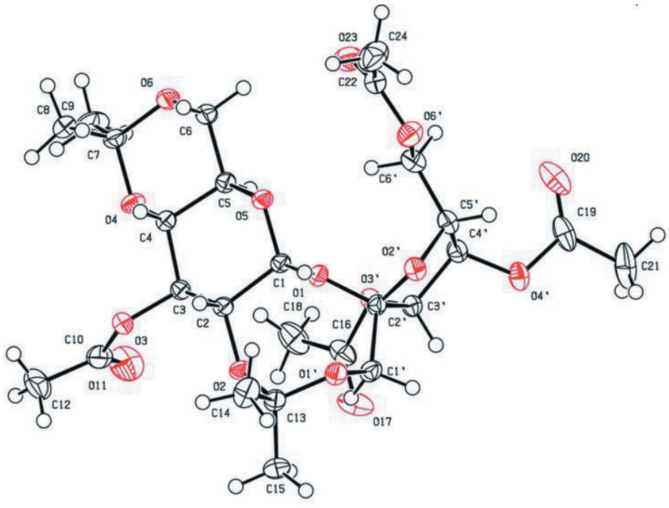


Figure 1
The molecular structure of the title compound with atom labelling [same as employed by Drew *et al.* (1979)]. Displacement ellipsoids are drawn at the 50% probability level.

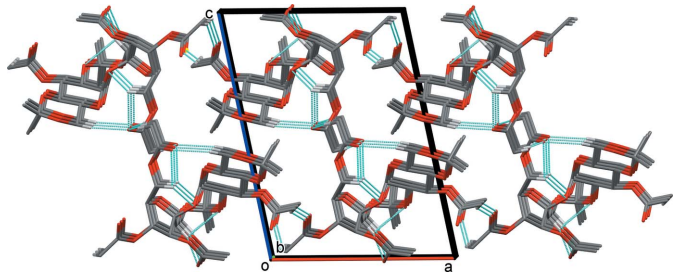


Figure 2
A view along the *b* axis of the crystal packing of the title compound. The hydrogen bonds (Table 1) are shown as dashed lines. For clarity, only the H atoms involved in the intra- and intermolecular hydrogen bonds have been included.

Synthesis and crystallization

Following a literature procedure (Khan and Mufti), 2,2-dimethoxypropane (25.0 ml, 204 mmol) was added, in one portion, to a magnetically stirred solution of sucrose (4.96 g, 14.5 mmol) and *p*-toluenesulfonic acid (514 mg, 3.00 mmol) in dimethylformamide (DMF, 250 ml) maintained at 295 K. The ensuing mixture was left to stir for 24 h before being treated with sodium bicarbonate (5 ml of a saturated aqueous solution) and the DMF then removed by distillation under reduced pressure (323 K at 15 mm Hg). The yellow gum thus obtained was dissolved in pyridine (80 ml) and the solution so formed treated, in one portion, with acetic anhydride (35.0 ml, 317 mmol). The reaction mixture was stirred at 295 K for 24 h then concentrated by removal of the pyridine through co-distillation with toluene and so leaving a golden-coloured residue. This residue was subjected to flash chromatography (silica, 1:1 *v*/*v* ethyl acetate/40–60 petroleum spirits elution) and concentration of the relevant fractions (R_f = 0.3 in 2:3 *v*/*v* ethyl acetate/40–60 petroleum spirits) afforded the title compound (878 mg, 10%) as colourless needles. A small sample of this material was recrystallized by vapour diffusion (using diethyl ether and 40–60 petroleum spirits) to afford single crystals suitable for X-ray diffraction analysis, m.p. 406-408 K [lit. (Fanton *et al.*, 1981) m.p. 409–410 K], $[\alpha]_D$ +10.3 (*c* = 1.0, chloroform) {lit. (Fanton *et al.*, 1981) $[\alpha]_D$ +13 (*c* = 1, chloroform)}.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.10 (*d*, *J* = 3.5 Hz, 1H), 5.33–5.28 (complex *m*, 1H), 5.21 (*t*, *J* = 9.5 Hz, 1H), 5.15 (*d*, *J* = 6.1 Hz, 1H), 4.40 (*q*, *J* = 9.2 Hz, 1H), 4.31–4.21 (complex *m*, 2H), 4.03 (*d*, *J* = 12.5 Hz, 1H), 3.96 (*m*, 1H), 3.90–3.77 (complex *m*, 2H), 3.66 (*m*, 2H), 3.51 (*d*, *J* = 12.5 Hz, 1H), 2.23 (*s*, 3H), 2.09 (*s*, 3H), 2.05 (*s*, 3H), 2.04 (*s*, 3H), 1.46 (*s*, 3H), 1.45 (*s*, 3H), 1.40 (*s*, 3H), 1.26 (*s*, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.0, 171.6, 171.2, 170.0, 105.2, 101.6, 92.5, 90.7, 80.2, 79.5, 78.4, 76.3, 73.6, 70.6, 69.7, 64.5, 64.3, 62.7, 25.6, 24.1, 21.3, 21.1, 20.9 (4), 20 (9), 20.8 (one signal obscured or overlapping); IR (film) ν_{max} 2998, 2939, 1742, 1371, 1221, 1151, 1131, 1069, 1047, 1034, 1017, 945, 894, 856, 735 cm^{-1}; LRMS (ESI, +ve) *m*/*z* 613 [(*M* + Na)$^+$, 100%]; HRMS (ESI, +ve) calculated for $C_{26}H_{38}O_{15}Na$ [(*M* + Na)$^+$] 613.2103, found [(*M* + Na)$^+$] 613.2103.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{26}H_{38}O_{15}$
M_r	590.56
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	150
a, b, c (Å)	11.1629 (2), 8.7778 (1), 15.3748 (3)
β (°)	101.558 (2)
V (Å^3)	1475.96 (4)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.94
Crystal size (mm)	0.26 × 0.2 × 0.15
Data collection	
Diffractometer	Rigaku Oxfird Diffraction SuperNova, Dual, Cu at home/near, EosS2
Absorption correction	Integration (*CrysAlis PRO*; Rigaku OD, 2018)
T_{min}, T_{max}	0.920, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10371, 5289, 5113
R_{int}	0.026
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.623
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.098, 1.02
No. of reflections	5289
No. of parameters	378
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.29
Absolute structure	Flack x determined using 2026 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.05 (8)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009), *Mercury* (Macrae *et al.*, 2008), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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We thank Mr Han-Lin Huang and Mr Huai-Yi Xu (Guangzhou Cardlo Biochemical Technology Company Ltd) for useful comments and the facilitation of this research. We thank Dr Ping Lan and Professor Yong Wang (Jinan University, Guangzhou) for assistance in manifold ways.

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full crystallographic data

IUCrData (2019). **4**, x190986 [https://doi.org/10.1107/S2414314619009866]

(2*S*,3*S*,4*R*,4a′*R*,5*R*,5a′*R*,11a′*R*,12′*S*,12a′*R*)-5-(Acetoxymethyl)-2′,2′,10′,10′-tetramethyloctahydro-3*H*,8′*H*-spiro[furan-2,7′-[1,3]dioxino[4′,5′:5,6]pyrano[3,2-*d*][1,3,6]trioxocine]-3,4,12′-triyl triacetate

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(2*S*,3*S*,4*R*,4a′*R*,5*R*,5a′*R*,11a′*R*,12′*S*,12a′*R*)-5-(Acetoxymethyl)-2′,2′,10′,10′-tetramethyloctahydro-3*H*,8′*H*-spiro[furan-2,7′-[1,3]dioxino[4′,5′:5,6]pyrano[3,2-*d*][1,3,6]trioxocine]-3,4,12′-triyl triacetate

Crystal data

$C_{26}H_{38}O_{15}$
M_r = 590.56
Monoclinic, $P2_1$
a = 11.1629 (2) Å
b = 8.7778 (1) Å
c = 15.3748 (3) Å
β = 101.558 (2)°
V = 1475.96 (4) Å^3
Z = 2
$F(000)$ = 628
D_x = 1.329 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 6926 reflections
θ = 5.0–73.6°
μ = 0.94 mm^{-1}
T = 150 K
Block, colourless
0.26 × 0.2 × 0.15 mm

Data collection

Rigaku Oxfird Diffraction SuperNova, Dual, Cu at home/near, EosS2 diffractometer
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 8.1297 pixels mm^{-1}
ω scans
Absorption correction: integration (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.920, T_{max} = 1.000
10371 measured reflections
5289 independent reflections
5113 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 73.7°, θ_{min} = 4.0°
h = −13→11
k = −10→10
l = −18→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.098
S = 1.02
5289 reflections
378 parameters
1 restraint
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2 + 0.0718P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack x determined using 2026 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.05 (8)

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. The H atoms attached to the carbon atoms were introduced in calculated positions and treated as riding: C—H = 0.96–0.98 Å with U_{iso}(H) = 1.5U_{eq}(C-methyl) and 1.2U_{eq}(C) for other H atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.66805 (13)	0.44758 (16)	0.23465 (9)	0.0194 (4)
O1′	0.59371 (14)	0.76853 (18)	0.21908 (10)	0.0230 (4)
O2	0.79603 (14)	0.70874 (17)	0.21177 (10)	0.0212 (4)
O2′	0.46513 (13)	0.48539 (18)	0.24952 (10)	0.0227 (4)
O3	1.03877 (13)	0.61194 (18)	0.27262 (10)	0.0231 (4)
O3′	0.60065 (13)	0.29538 (19)	0.09135 (10)	0.0233 (4)
O4	1.05077 (13)	0.29710 (18)	0.32930 (10)	0.0226 (4)
O4′	0.30285 (14)	0.2773 (2)	0.09726 (11)	0.0293 (5)
O5	0.76401 (13)	0.42594 (17)	0.38307 (10)	0.0202 (4)
O6	0.98841 (14)	0.11966 (18)	0.42674 (11)	0.0258 (4)
O6′	0.47916 (15)	0.2898 (2)	0.40486 (11)	0.0300 (5)
O11	1.0621 (2)	0.5190 (3)	0.14086 (14)	0.0498 (7)
O17	0.6223 (2)	0.4819 (3)	−0.00490 (13)	0.0438 (6)
O20	0.2460 (2)	0.0736 (3)	0.16760 (14)	0.0447 (6)
O23	0.61677 (18)	0.1156 (2)	0.46666 (14)	0.0417 (6)
C1	0.72380 (18)	0.5291 (2)	0.31234 (13)	0.0180 (5)
C1′	0.5461 (2)	0.6575 (2)	0.15460 (14)	0.0239 (6)
C2	0.83133 (18)	0.6213 (2)	0.29037 (13)	0.0181 (5)
C2′	0.54969 (17)	0.4956 (2)	0.19222 (13)	0.0195 (5)
C3	0.93376 (17)	0.5178 (2)	0.27383 (13)	0.0187 (5)
C3′	0.50360 (18)	0.3786 (2)	0.11750 (14)	0.0210 (5)
C4	0.96673 (18)	0.4064 (2)	0.34956 (13)	0.0188 (5)
C4′	0.42079 (18)	0.2699 (3)	0.15538 (14)	0.0232 (6)
C5	0.85467 (18)	0.3224 (2)	0.36547 (14)	0.0195 (5)
C5′	0.41843 (19)	0.3324 (3)	0.24868 (14)	0.0235 (6)
C6	0.8952 (2)	0.2196 (3)	0.44536 (15)	0.0238 (6)
C6′	0.4918 (2)	0.2320 (3)	0.31931 (15)	0.0264 (6)
C7	1.0907 (2)	0.1927 (3)	0.40087 (14)	0.0247 (6)
C8	1.1721 (2)	0.2719 (3)	0.47857 (16)	0.0332 (7)
C9	1.1574 (3)	0.0669 (3)	0.36347 (19)	0.0400 (8)
C10	1.0970 (2)	0.5994 (3)	0.20445 (16)	0.0308 (7)
C12	1.2079 (3)	0.6986 (4)	0.2192 (2)	0.0475 (10)
C13	0.71093 (19)	0.8288 (2)	0.21280 (15)	0.0238 (6)
C14	0.7437 (2)	0.9332 (3)	0.29265 (19)	0.0354 (7)
C15	0.7081 (2)	0.9135 (3)	0.12672 (18)	0.0346 (7)
C16	0.6578 (2)	0.3652 (3)	0.03229 (16)	0.0303 (7)
C18	0.7696 (3)	0.2792 (4)	0.0219 (2)	0.0444 (9)
C19	0.2232 (2)	0.1664 (3)	0.10960 (19)	0.0351 (8)

C21	0.1074 (3)	0.1793 (4)	0.0416 (3)	0.0563 (10)
C22	0.5493 (2)	0.2184 (3)	0.47446 (16)	0.0305 (7)
C24	0.5324 (3)	0.2839 (4)	0.56148 (17)	0.0451 (9)
H1	0.66400	0.59930	0.32900	0.0220*
H1′A	0.59270	0.65990	0.10770	0.0290*
H2	0.86320	0.68960	0.34000	0.0220*
H1′B	0.46220	0.68350	0.12850	0.0290*
H3	0.90920	0.46360	0.21730	0.0220*
H3′	0.45590	0.43140	0.06580	0.0250*
H6′A	0.46210	0.12800	0.31200	0.0320*
H4	1.00430	0.46160	0.40360	0.0230*
H4′	0.45320	0.16580	0.15870	0.0280*
H6′B	0.57720	0.23260	0.31460	0.0320*
H5	0.82070	0.26050	0.31330	0.0230*
H5′	0.33360	0.33590	0.25680	0.0280*
H6A	0.92700	0.28040	0.49760	0.0290*
H6B	0.82640	0.16080	0.45670	0.0290*
H8A	1.23400	0.32900	0.45770	0.0500*
H8B	1.21000	0.19720	0.52080	0.0500*
H8C	1.12360	0.33970	0.50640	0.0500*
H9A	1.10380	0.02140	0.31360	0.0600*
H9B	1.18300	−0.00900	0.40830	0.0600*
H9C	1.22770	0.10820	0.34470	0.0600*
H12A	1.18400	0.80200	0.20450	0.0710*
H12B	1.26230	0.66470	0.18210	0.0710*
H12C	1.24860	0.69300	0.28030	0.0710*
H14A	0.73220	0.88010	0.34500	0.0530*
H14B	0.69200	1.02160	0.28380	0.0530*
H14C	0.82760	0.96410	0.29970	0.0530*
H15A	0.78360	0.96760	0.13000	0.0520*
H15B	0.64130	0.98450	0.11710	0.0520*
H15C	0.69750	0.84220	0.07850	0.0520*
H18A	0.82900	0.28350	0.07640	0.0670*
H18B	0.80310	0.32390	−0.02490	0.0670*
H18C	0.74830	0.17490	0.00760	0.0670*
H21A	0.07330	0.27910	0.04430	0.0840*
H21B	0.05000	0.10430	0.05330	0.0840*
H21C	0.12450	0.16260	−0.01640	0.0840*
H24A	0.60370	0.34140	0.58760	0.0680*
H24B	0.52070	0.20270	0.60080	0.0680*
H24C	0.46210	0.34930	0.55170	0.0680*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0172 (6)	0.0165 (7)	0.0227 (7)	0.0008 (5)	−0.0002 (5)	−0.0012 (5)
O1′	0.0229 (7)	0.0158 (7)	0.0291 (7)	0.0017 (6)	0.0022 (6)	−0.0014 (6)
O2	0.0232 (7)	0.0156 (7)	0.0239 (7)	0.0026 (6)	0.0023 (5)	0.0035 (5)

O2′	0.0216 (7)	0.0198 (8)	0.0273 (7)	−0.0001 (6)	0.0061 (5)	−0.0025 (6)
O3	0.0203 (7)	0.0241 (8)	0.0251 (7)	−0.0018 (6)	0.0053 (5)	0.0021 (6)
O3′	0.0244 (7)	0.0231 (8)	0.0236 (7)	−0.0032 (6)	0.0076 (6)	−0.0003 (6)
O4	0.0247 (7)	0.0219 (8)	0.0216 (7)	0.0079 (6)	0.0053 (5)	0.0048 (6)
O4′	0.0220 (7)	0.0296 (9)	0.0348 (8)	−0.0077 (7)	0.0024 (6)	−0.0061 (7)
O5	0.0198 (6)	0.0190 (7)	0.0217 (7)	0.0031 (5)	0.0039 (5)	0.0031 (5)
O6	0.0242 (7)	0.0188 (8)	0.0347 (8)	0.0043 (6)	0.0067 (6)	0.0072 (6)
O6′	0.0287 (8)	0.0359 (9)	0.0256 (8)	0.0057 (7)	0.0056 (6)	0.0014 (7)
O11	0.0547 (12)	0.0615 (15)	0.0396 (10)	−0.0008 (11)	0.0250 (9)	−0.0083 (10)
O17	0.0611 (12)	0.0369 (11)	0.0387 (10)	−0.0029 (10)	0.0230 (9)	0.0094 (8)
O20	0.0497 (11)	0.0409 (11)	0.0503 (11)	−0.0190 (9)	0.0264 (9)	−0.0098 (9)
O23	0.0404 (10)	0.0386 (11)	0.0480 (11)	0.0102 (9)	0.0137 (8)	0.0165 (9)
C1	0.0197 (8)	0.0137 (9)	0.0194 (9)	0.0002 (7)	0.0009 (7)	−0.0001 (7)
C1′	0.0241 (10)	0.0187 (11)	0.0260 (10)	0.0000 (8)	−0.0017 (8)	0.0017 (8)
C2	0.0203 (9)	0.0137 (9)	0.0191 (9)	−0.0008 (7)	0.0010 (7)	−0.0006 (7)
C2′	0.0172 (8)	0.0171 (10)	0.0230 (9)	−0.0004 (7)	0.0010 (7)	−0.0005 (8)
C3	0.0196 (9)	0.0163 (9)	0.0197 (9)	−0.0009 (8)	0.0030 (7)	−0.0002 (7)
C3′	0.0201 (9)	0.0200 (10)	0.0218 (9)	−0.0027 (8)	0.0014 (7)	−0.0004 (8)
C4	0.0187 (9)	0.0181 (10)	0.0188 (9)	0.0009 (7)	0.0021 (7)	−0.0005 (7)
C4′	0.0206 (9)	0.0219 (11)	0.0270 (10)	−0.0024 (8)	0.0046 (8)	−0.0011 (8)
C5	0.0200 (9)	0.0149 (10)	0.0224 (9)	0.0007 (7)	0.0016 (7)	−0.0007 (7)
C5′	0.0216 (9)	0.0218 (11)	0.0280 (11)	−0.0032 (8)	0.0073 (8)	−0.0023 (8)
C6	0.0210 (9)	0.0214 (11)	0.0291 (10)	0.0026 (8)	0.0055 (8)	0.0066 (8)
C6′	0.0289 (11)	0.0233 (11)	0.0293 (11)	−0.0011 (8)	0.0111 (8)	0.0008 (8)
C7	0.0228 (9)	0.0256 (11)	0.0263 (10)	0.0066 (9)	0.0061 (8)	0.0095 (9)
C8	0.0250 (10)	0.0414 (14)	0.0302 (11)	−0.0009 (10)	−0.0016 (8)	0.0134 (11)
C9	0.0433 (14)	0.0352 (14)	0.0456 (14)	0.0212 (12)	0.0189 (12)	0.0131 (12)
C10	0.0282 (11)	0.0326 (13)	0.0342 (12)	0.0082 (9)	0.0128 (9)	0.0089 (10)
C12	0.0312 (13)	0.0523 (18)	0.0643 (19)	−0.0029 (13)	0.0224 (12)	0.0116 (15)
C13	0.0222 (9)	0.0144 (9)	0.0328 (11)	0.0012 (8)	0.0008 (8)	0.0017 (8)
C14	0.0350 (12)	0.0178 (11)	0.0483 (14)	0.0034 (9)	−0.0040 (10)	−0.0093 (10)
C15	0.0332 (11)	0.0249 (12)	0.0439 (14)	0.0042 (10)	0.0034 (10)	0.0152 (10)
C16	0.0367 (12)	0.0307 (13)	0.0261 (11)	−0.0106 (10)	0.0126 (9)	−0.0043 (9)
C18	0.0444 (15)	0.0461 (17)	0.0505 (15)	−0.0060 (13)	0.0285 (12)	−0.0051 (14)
C19	0.0281 (11)	0.0363 (15)	0.0447 (14)	−0.0138 (10)	0.0164 (10)	−0.0206 (12)
C21	0.0255 (12)	0.059 (2)	0.082 (2)	−0.0139 (13)	0.0054 (14)	−0.0300 (19)
C22	0.0220 (10)	0.0339 (13)	0.0348 (12)	−0.0023 (9)	0.0040 (9)	0.0080 (10)
C24	0.0412 (14)	0.062 (2)	0.0297 (12)	0.0069 (14)	0.0016 (10)	0.0038 (13)

Geometric parameters (Å, º)

O1—C1	1.424 (2)	C16—C18	1.494 (4)
O1—C2′	1.416 (2)	C19—C21	1.495 (5)
O1′—C1′	1.416 (2)	C22—C24	1.502 (4)
O1′—C13	1.433 (3)	C1—H1	0.9800
O2—C2	1.419 (2)	C1′—H1′A	0.9700
O2—C13	1.421 (2)	C1′—H1′B	0.9700
O2′—C2′	1.417 (2)	C2—H2	0.9800

O2′—C5′	1.440 (3)	C3—H3	0.9800
O3—C3	1.437 (2)	C3′—H3′	0.9800
O3—C10	1.344 (3)	C4—H4	0.9800
O3′—C3′	1.430 (3)	C4′—H4′	0.9800
O3′—C16	1.356 (3)	C5—H5	0.9800
O4—C4	1.419 (2)	C5′—H5′	0.9800
O4—C7	1.433 (3)	C6—H6A	0.9700
O4′—C4′	1.437 (3)	C6—H6B	0.9700
O4′—C19	1.357 (3)	C6′—H6′A	0.9700
O5—C1	1.417 (2)	C6′—H6′B	0.9700
O5—C5	1.426 (2)	C8—H8A	0.9600
O6—C6	1.433 (3)	C8—H8B	0.9600
O6—C7	1.434 (3)	C8—H8C	0.9600
O6′—C6′	1.443 (3)	C9—H9A	0.9600
O6′—C22	1.347 (3)	C9—H9B	0.9600
O11—C10	1.205 (3)	C9—H9C	0.9600
O17—C16	1.201 (4)	C12—H12A	0.9600
O20—C19	1.197 (4)	C12—H12B	0.9600
O23—C22	1.197 (3)	C12—H12C	0.9600
C1—C2	1.540 (3)	C14—H14A	0.9600
C1′—C2′	1.532 (3)	C14—H14B	0.9600
C2—C3	1.521 (3)	C14—H14C	0.9600
C2′—C3′	1.550 (3)	C15—H15A	0.9600
C3—C4	1.509 (3)	C15—H15B	0.9600
C3′—C4′	1.523 (3)	C15—H15C	0.9600
C4—C5	1.514 (3)	C18—H18A	0.9600
C4′—C5′	1.541 (3)	C18—H18B	0.9600
C5—C6	1.518 (3)	C18—H18C	0.9600
C5′—C6′	1.507 (3)	C21—H21A	0.9600
C7—C8	1.517 (3)	C21—H21B	0.9600
C7—C9	1.509 (4)	C21—H21C	0.9600
C10—C12	1.493 (4)	C24—H24A	0.9600
C13—C14	1.517 (3)	C24—H24B	0.9600
C13—C15	1.513 (3)	C24—H24C	0.9600
C1—O1—C2′	116.59 (15)	C3—C2—H2	109.00
C1′—O1′—C13	115.27 (16)	O3—C3—H3	111.00
C2—O2—C13	117.63 (16)	C2—C3—H3	111.00
C2′—O2′—C5′	109.79 (15)	C4—C3—H3	111.00
C3—O3—C10	119.09 (17)	O3′—C3′—H3′	109.00
C3′—O3′—C16	116.24 (17)	C2′—C3′—H3′	109.00
C4—O4—C7	112.42 (16)	C4′—C3′—H3′	109.00
C4′—O4′—C19	115.15 (19)	O4—C4—H4	109.00
C1—O5—C5	112.92 (15)	C3—C4—H4	109.00
C6—O6—C7	115.61 (18)	C5—C4—H4	109.00
C6′—O6′—C22	114.40 (19)	O4′—C4′—H4′	111.00
O1—C1—O5	109.91 (14)	C3′—C4′—H4′	111.00
O1—C1—C2	107.99 (15)	C5′—C4′—H4′	111.00

O5—C1—C2	111.29 (16)	O5—C5—H5	110.00
O1′—C1′—C2′	113.36 (17)	C4—C5—H5	110.00
O2—C2—C1	112.04 (16)	C6—C5—H5	110.00
O2—C2—C3	105.69 (16)	O2′—C5′—H5′	109.00
C1—C2—C3	111.58 (15)	C4′—C5′—H5′	109.00
O1—C2′—O2′	111.79 (15)	C6′—C5′—H5′	109.00
O1—C2′—C1′	113.68 (16)	O6—C6—H6A	110.00
O1—C2′—C3′	106.55 (15)	O6—C6—H6B	110.00
O2′—C2′—C1′	108.85 (16)	C5—C6—H6A	110.00
O2′—C2′—C3′	104.79 (15)	C5—C6—H6B	110.00
C1′—C2′—C3′	110.80 (16)	H6A—C6—H6B	108.00
O3—C3—C2	107.52 (14)	O6′—C6′—H6′A	110.00
O3—C3—C4	108.01 (16)	O6′—C6′—H6′B	110.00
C2—C3—C4	109.48 (16)	C5′—C6′—H6′A	110.00
O3′—C3′—C2′	112.91 (16)	C5′—C6′—H6′B	110.00
O3′—C3′—C4′	109.86 (16)	H6′A—C6′—H6′B	108.00
C2′—C3′—C4′	105.57 (17)	C7—C8—H8A	110.00
O4—C4—C3	109.79 (16)	C7—C8—H8B	109.00
O4—C4—C5	108.07 (15)	C7—C8—H8C	109.00
C3—C4—C5	111.03 (16)	H8A—C8—H8B	110.00
O4′—C4′—C3′	106.53 (18)	H8A—C8—H8C	109.00
O4′—C4′—C5′	112.23 (17)	H8B—C8—H8C	109.00
C3′—C4′—C5′	104.94 (19)	C7—C9—H9A	109.00
O5—C5—C4	111.17 (15)	C7—C9—H9B	109.00
O5—C5—C6	109.46 (17)	C7—C9—H9C	110.00
C4—C5—C6	107.36 (17)	H9A—C9—H9B	109.00
O2′—C5′—C4′	105.47 (18)	H9A—C9—H9C	109.00
O2′—C5′—C6′	113.30 (18)	H9B—C9—H9C	110.00
C4′—C5′—C6′	110.8 (2)	C10—C12—H12A	109.00
O6—C6—C5	108.23 (17)	C10—C12—H12B	109.00
O6′—C6′—C5′	108.24 (19)	C10—C12—H12C	110.00
O4—C7—O6	110.89 (17)	H12A—C12—H12B	109.00
O4—C7—C8	110.9 (2)	H12A—C12—H12C	109.00
O4—C7—C9	106.07 (18)	H12B—C12—H12C	110.00
O6—C7—C8	111.79 (18)	C13—C14—H14A	110.00
O6—C7—C9	105.1 (2)	C13—C14—H14B	109.00
C8—C7—C9	111.9 (2)	C13—C14—H14C	109.00
O3—C10—O11	123.5 (2)	H14A—C14—H14B	109.00
O3—C10—C12	110.5 (2)	H14A—C14—H14C	109.00
O11—C10—C12	126.0 (2)	H14B—C14—H14C	109.00
O1′—C13—O2	110.40 (15)	C13—C15—H15A	109.00
O1′—C13—C14	104.12 (17)	C13—C15—H15B	109.00
O1′—C13—C15	112.41 (18)	C13—C15—H15C	109.00
O2—C13—C14	113.81 (18)	H15A—C15—H15B	109.00
O2—C13—C15	104.57 (18)	H15A—C15—H15C	110.00
C14—C13—C15	111.75 (18)	H15B—C15—H15C	109.00
O3′—C16—O17	123.3 (2)	C16—C18—H18A	109.00
O3′—C16—C18	111.0 (2)	C16—C18—H18B	109.00

O17—C16—C18	125.7 (2)	C16—C18—H18C	109.00
O4′—C19—O20	123.0 (2)	H18A—C18—H18B	110.00
O4′—C19—C21	110.3 (2)	H18A—C18—H18C	109.00
O20—C19—C21	126.7 (3)	H18B—C18—H18C	110.00
O6′—C22—O23	123.3 (2)	C19—C21—H21A	110.00
O6′—C22—C24	111.9 (2)	C19—C21—H21B	109.00
O23—C22—C24	124.9 (2)	C19—C21—H21C	109.00
O1—C1—H1	109.00	H21A—C21—H21B	109.00
O5—C1—H1	109.00	H21A—C21—H21C	110.00
C2—C1—H1	109.00	H21B—C21—H21C	109.00
O1′—C1′—H1′A	109.00	C22—C24—H24A	109.00
O1′—C1′—H1′B	109.00	C22—C24—H24B	109.00
C2′—C1′—H1′A	109.00	C22—C24—H24C	110.00
C2′—C1′—H1′B	109.00	H24A—C24—H24B	109.00
H1′A—C1′—H1′B	108.00	H24A—C24—H24C	110.00
O2—C2—H2	109.00	H24B—C24—H24C	109.00
C1—C2—H2	109.00		

C2′—O1—C1—O5	127.72 (16)	C6—O6—C7—C8	72.3 (2)
C2′—O1—C1—C2	−110.71 (17)	C6—O6—C7—C9	−166.16 (19)
C1—O1—C2′—O2′	−59.4 (2)	C22—O6′—C6′—C5′	−174.31 (19)
C1—O1—C2′—C1′	64.3 (2)	C6′—O6′—C22—O23	0.0 (3)
C1—O1—C2′—C3′	−173.37 (15)	C6′—O6′—C22—C24	179.8 (2)
C13—O1′—C1′—C2′	107.1 (2)	O1—C1—C2—O2	50.69 (19)
C1′—O1′—C13—O2	−53.1 (2)	O1—C1—C2—C3	−67.60 (19)
C1′—O1′—C13—C14	−175.61 (17)	O5—C1—C2—O2	171.41 (15)
C1′—O1′—C13—C15	63.3 (2)	O5—C1—C2—C3	53.1 (2)
C13—O2—C2—C1	65.6 (2)	O1′—C1′—C2′—O1	−56.7 (2)
C13—O2—C2—C3	−172.65 (16)	O1′—C1′—C2′—O2′	68.6 (2)
C2—O2—C13—O1′	−67.5 (2)	O1′—C1′—C2′—C3′	−176.64 (17)
C2—O2—C13—C14	49.1 (2)	O2—C2—C3—O3	69.91 (18)
C2—O2—C13—C15	171.34 (17)	O2—C2—C3—C4	−172.99 (15)
C5′—O2′—C2′—O1	−83.67 (18)	C1—C2—C3—O3	−168.07 (15)
C5′—O2′—C2′—C1′	149.92 (16)	C1—C2—C3—C4	−51.0 (2)
C5′—O2′—C2′—C3′	31.35 (19)	O1—C2′—C3′—O3′	−21.2 (2)
C2′—O2′—C5′—C4′	−30.2 (2)	O1—C2′—C3′—C4′	98.89 (18)
C2′—O2′—C5′—C6′	91.2 (2)	O2′—C2′—C3′—O3′	−139.76 (16)
C10—O3—C3—C2	−129.51 (19)	O2′—C2′—C3′—C4′	−19.7 (2)
C10—O3—C3—C4	112.4 (2)	C1′—C2′—C3′—O3′	102.99 (19)
C3—O3—C10—O11	4.3 (4)	C1′—C2′—C3′—C4′	−136.97 (17)
C3—O3—C10—C12	−176.0 (2)	O3—C3—C4—O4	−70.72 (19)
C16—O3′—C3′—C2′	−82.6 (2)	O3—C3—C4—C5	169.85 (15)
C16—O3′—C3′—C4′	159.83 (18)	C2—C3—C4—O4	172.49 (15)
C3′—O3′—C16—O17	−8.5 (3)	C2—C3—C4—C5	53.1 (2)
C3′—O3′—C16—C18	170.6 (2)	O3′—C3′—C4′—O4′	−116.56 (19)
C7—O4—C4—C3	176.87 (16)	O3′—C3′—C4′—C5′	124.25 (18)
C7—O4—C4—C5	−61.9 (2)	C2′—C3′—C4′—O4′	121.41 (18)
C4—O4—C7—O6	55.2 (2)	C2′—C3′—C4′—C5′	2.2 (2)

C4—O4—C7—C8	−69.6 (2)	O4—C4—C5—O5	−177.86 (15)
C4—O4—C7—C9	168.74 (19)	O4—C4—C5—C6	62.5 (2)
C19—O4′—C4′—C3′	167.35 (19)	C3—C4—C5—O5	−57.4 (2)
C19—O4′—C4′—C5′	−78.3 (3)	C3—C4—C5—C6	−177.09 (16)
C4′—O4′—C19—O20	4.0 (4)	O4′—C4′—C5′—O2′	−99.5 (2)
C4′—O4′—C19—C21	−176.1 (2)	O4′—C4′—C5′—C6′	137.5 (2)
C5—O5—C1—O1	62.2 (2)	C3′—C4′—C5′—O2′	15.8 (2)
C5—O5—C1—C2	−57.4 (2)	C3′—C4′—C5′—C6′	−107.2 (2)
C1—O5—C5—C4	59.9 (2)	O5—C5—C6—O6	−178.50 (16)
C1—O5—C5—C6	178.32 (16)	C4—C5—C6—O6	−57.7 (2)
C7—O6—C6—C5	54.2 (2)	O2′—C5′—C6′—O6′	65.7 (2)
C6—O6—C7—O4	−52.0 (2)	C4′—C5′—C6′—O6′	−175.99 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1′	0.98	2.27	2.783 (2)	112
C1—H1···O2′	0.98	2.52	2.881 (3)	102
C1′—H1′*A*···O17	0.97	2.40	3.154 (3)	134
C3—H3···O11	0.98	2.31	2.721 (3)	104
C6′—H6′*B*···O1	0.97	2.57	3.189 (3)	122
C6—H6*B*···O23	0.97	2.41	3.318 (3)	156
C12—H12*A*···O20^{i}	0.96	2.58	3.432 (4)	148
C21—H21*A*···O11^{ii}	0.96	2.59	3.433 (4)	146
C24—H24*C*···O23^{iii}	0.96	2.49	3.339 (4)	147

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