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1-*O*-Acetyl-3,4,6-tri-*O*-benzyl-2-*C*-bromomethyl-2-deoxy-*α*-D-glucopyranose

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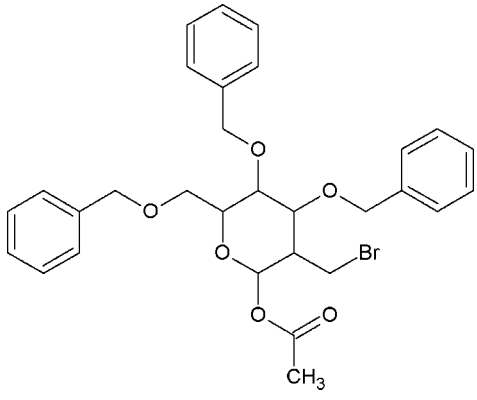
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.006 Å; R factor = 0.041; wR factor = 0.096; data-to-parameter ratio = 13.8.

In the title compound, $C_{30}H_{33}BrO_6$, the pyranose ring adopts a chair conformation. Two of the *O*-benzyl phenyl rings lie almost perpendicular to C/C/C/O plane formed by the ring atoms not attached to these *O*-benzyl phenyl rings, and form dihedral angles of 85.1 (2) and 64.6 (2)°, while the third *O*-benzyl phenyl ring is twisted so that it makes a dihedral angle 34.9 (2)° to this C/C/C/O plane. This twist is ascribed to the formation of an *S*(8) loop stabilized by a weak intramolecular C—H···O hydrogen bond.

Related literature

For background to derivatization of cyclopropyl carbohydrates, see: Halton & Harvey (2006); Beyer & Madsen (1998). For details of the synthesis of the title compound, see: Gammon *et al.* (2007). For ring puckering analysis, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{30}H_{33}BrO_6$
M_r = 569.47
Orthorhombic, $P2_12_12_1$
a = 5.5097 (4) Å
b = 19.9357 (11) Å
c = 24.4597 (16) Å
V = 2686.6 (3) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.43 mm^{-1}
T = 100 K
0.43 × 0.05 × 0.05 mm

Data collection

Bruker APEX DUO 4K-CCD diffractometer
Absorption correction: multi-scan (*SADABS*, Bruker, 2008)
T_{min} = 0.613, T_{max} = 0.753
51826 measured reflections
4637 independent reflections
4177 reflections with $I > 2\sigma(I)$
R_{int} = 0.097

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.096
S = 1.04
4637 reflections
335 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.04 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}
Absolute structure: Flack (1983), 1893 Friedel pairs
Flack parameter: −0.01 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C18—H18···O4	0.95	2.85	3.663 (7)	144

Data collection: *APEX2* (Bruker, 2011); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SUPERFLIP* (Palatinus & Chapuis, 2007); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.* 2009); software used to prepare material for publication: *OLEX2*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5273).

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1-*O*-Acetyl-3,4,6-tri-*O*-benzyl-2-*C*-bromomethyl-2-deoxy-*α*-D-glucopyranose

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S1. Comment

The derivatization of cyclopropyl carbohydrates remains a relatively small area of research and, of this work, only a small proportion is devoted to reactions of the cyclopropyl moiety (Halton *et al.*, 2006). Nucleophilic attack by a range of alcohols, including monosaccharides afforded 2-C-branched glycosides (Beyer *et al.*, 1998). The compound of interest is a useful glycosyl donor and a precursor for the extension of the C–2 side chain (Gammon *et al.*, 2007).

In the title compound the pyranose ring adopts a chair conformation with ring puckering parameters of q_2 = 0.0601 Å, q_3 = 0.5555 Å, Q = 0.5587 Å, θ = 6.17 ° and φ_2 = 280.0707° (see Cremer & Pople, 1975) (see Figure 1). The C10 – C15 and C25 – C30 rings, lie almost perpendicular to the C1 C3 C4 O1 plane of the pyranose ring, with dihedral angles of 85.1 (2) and 64.6 (2) ° respectively. The C17 – C22 phenyl ring is twisted to an almost parallel position to the C1 C3 C4 O1 plane with a dihedral angle of 34.9 (2)°. The twist is ascribed to the formation of an *S*(8) loop stabilized by weak intramolecular C–H···O hydrogen bond (see Figure 1; Table 1).

S2. Experimental

The cyclopropane, 3,4,6–Tri–O–benzyl–1,5–anhydro–2–deoxy–1,2– C–methylene–D–glycero–D–gulo–hexitol (120 mg, 0.28 mmol) was suspended in 1:1 mixture of water and 1,4–dioxane (2 mL), N-Bromosuccinamide(NBS) (163 mg, 0.84 mmol) was added, then stirring was allowed overnight at room temperature. The reaction mixture was diluted with 10% aqueous solution of sodium thiosulfate, $Na_2S_2O_3$, and the aqueous phase extracted with ethyl acetate; the combined organic extracts were dried over $MgSO_4$, and concentrated *in vacuo*. The residue was acetylated without purification, acetic anhydride (0.5 ml) in pyridine (1 mL) at room temperature. After 1 h, water was added to the reaction, and the mixture was extracted with dichloromethane. The combined extracts were washed with 1*M* HCl and saturated aqueous $NaHCO_3$, dried over $MgSO_4$, concentrated under reduced pressure, and purified by flash chromatography to afford the title compound as a white solid.

Analytical data: mp: 115 – 117 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): σ 7.45 – 7.10 (m, 15H, Aromatic), 6.39 (d, *J* = 2.4Hz, 1H, H-1), 4.92 (d, *J* = 11.2Hz, 1H, C*H*$_2$Ph), 4.78 (d, *J* = 10.8Hz, 1H, C*H*$_2$Ph), 4.70 – 4.40 (m, 4H, C*H*$_2$Ph), 3.90 – 3.55 (m , 6H, H–3, H–4, H–5, H–6$_a$, H–6$_b$, H–7$_a$), 3.12 (t, *J* = 10.6Hz, 1H, H7$_b$), 2.45 – 2.25 (m, 1H, H–2), 2.09 (s, 3H, OCOC*H*$_3$); ^{13}C NMR ($CDCl_3$, 100 MHz): σ 168.9 (O*C*OCH$_3$), 137.8, 128.6, 128.5, 128.4, 128.0, 128.0, 127.9, 127.8, 127.8, 127.7(Aromatic), 92.1 (C–1), 79.7 (C–3), 78.8 (C–4), 75.4 (C–5), 75.0 (*C*H$_2$Ph), 73.6 (*C*H$_2$Ph), 73.3 (*C*H$_2$Ph), 68.1(C–6), 46.8 (C–2), 29.1(C–7), 20.9 (OCO*C*H$_3$).

S3. Refinement

All hydrogen atoms were positioned in geometrically idealized positions with C–H = 1.00 Å (methine), 0.99 Å (methylene), 0.95 Å aromatic and 0.98 Å (methyl). All hydrogen atoms were allowed to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}, except for the methyl where U_{iso}(H) = 1.5U_{eq} was utilized. The initial positions of methyl hydrogen atoms were

located from a Fourier difference map and refined as a fixed rotor.The D enantiomer refined to a final Flack parameter of -0.01 (2). The highest residual electron density of 1.04 e.Å^{-3} is 0.93 Å from Br1.

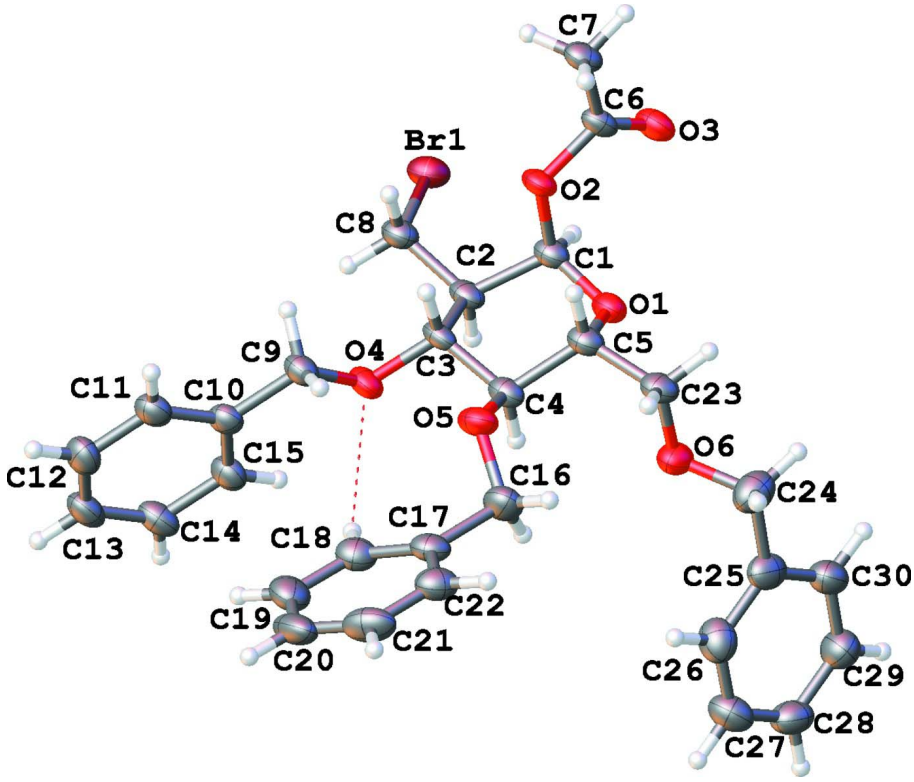

Figure 1

Crystal structure of the title compound with labeling and displacement ellipsoids drawn at the 50% probability level.

1-*O*-Acetyl-3,4,6-tri-*O*-benzyl-2-*C*-bromomethyl- 2-deoxy-*α*-*D*-glucopyranose

Crystal data

$C_{30}H_{33}BrO_6$
M_r = 569.47
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 5.5097 (4) Å
b = 19.9357 (11) Å
c = 24.4597 (16) Å
V = 2686.6 (3) Å^3
Z = 4
$F(000)$ = 1184
D_x = 1.408 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9957 reflections
θ = 4.2–65.2°
μ = 2.43 mm^{-1}
T = 100 K
Needle, colourless
0.43 × 0.05 × 0.05 mm

Data collection

Bruker APEX DUO 4K-CCD diffractometer
Radiation source: fine-focus sealed tube
Incoatec Quazar Multilayer Mirror monochromator
Detector resolution: 8.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*, Bruker, 2008)
T_{min} = 0.613, T_{max} = 0.753
51826 measured reflections
4637 independent reflections
4177 reflections with $I > 2\sigma(I)$
R_{int} = 0.097
θ_{max} = 66.2°, θ_{min} = 4.2°
h = −6→5
k = −23→23
l = −28→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.096$
$S = 1.04$
4637 reflections
335 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 1.595P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.04$ e Å^{-3}
$\Delta\rho_{min} = -0.61$ e Å^{-3}
Absolute structure: Flack (1983), 1893 Friedel pairs
Absolute structure parameter: −0.01 (2)

Special details

Experimental. Absorption correction: SADABS-2008/1 (Bruker,2008) was used for absorption correction. wR2(int) was 0.1272 before and 0.0933 after correction. The Ratio of minimum to maximum transmission is 0.8137. The $\lambda/2$ correction factor is 0.0015.
The intensity data was collected on a Bruker Apex DUO 4 K CCD diffractometer using an exposure time of 5 s/frame. A total of 4554 frames were collected with a frame width of 1° covering up to $\theta = 66.16°$ with 99.6% completeness accomplished.
Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
Br1	1.52718 (7)	0.657227 (17)	0.318194 (16)	0.03952 (13)
O1	1.1736 (6)	0.61572 (12)	0.48483 (10)	0.0340 (6)
O2	1.0466 (5)	0.68570 (10)	0.41379 (9)	0.0326 (6)
O3	1.2526 (6)	0.76628 (11)	0.45934 (11)	0.0400 (7)
O4	1.0000 (6)	0.48064 (10)	0.36332 (9)	0.0351 (6)
O5	0.7051 (6)	0.48859 (11)	0.45817 (11)	0.0392 (7)
O6	1.0735 (6)	0.52549 (12)	0.57192 (10)	0.0396 (7)
C1	1.2250 (8)	0.63710 (16)	0.43169 (15)	0.0316 (9)
H1	1.3896	0.6582	0.431	0.038*
C2	1.2183 (8)	0.57915 (16)	0.39091 (15)	0.0330 (9)
H2	1.3572	0.5485	0.3992	0.04*
C3	0.9832 (8)	0.53928 (14)	0.39714 (13)	0.0297 (8)
H3	0.8448	0.5675	0.384	0.036*
C4	0.9363 (7)	0.51944 (16)	0.45609 (14)	0.0319 (8)
H4	1.0638	0.4873	0.4688	0.038*
C5	0.9433 (8)	0.58369 (16)	0.49090 (14)	0.0338 (9)
H5	0.8151	0.6149	0.4771	0.041*
C6	1.0804 (8)	0.74997 (16)	0.43301 (15)	0.0312 (9)

C7	0.8758 (8)	0.79337 (17)	0.41525 (16)	0.0366 (10)
H7A	0.9038	0.8394	0.4279	0.055*
H7B	0.7239	0.7765	0.4309	0.055*
H7C	0.8646	0.7929	0.3753	0.055*
C8	1.2419 (8)	0.60206 (17)	0.33175 (15)	0.0375 (10)
H8A	1.0952	0.6279	0.3217	0.045*
H8B	1.249	0.562	0.3078	0.045*
C9	0.7950 (8)	0.46920 (17)	0.33029 (15)	0.0355 (9)
H9A	0.7645	0.5089	0.3069	0.043*
H9B	0.6505	0.462	0.3536	0.043*
C10	0.8379 (8)	0.40787 (17)	0.29466 (15)	0.0325 (9)
C11	0.6658 (8)	0.39109 (18)	0.25596 (15)	0.0384 (10)
H11	0.5251	0.4181	0.2515	0.046*
C12	0.6995 (9)	0.3342 (2)	0.22338 (16)	0.0443 (10)
H12	0.5808	0.3224	0.1969	0.053*
C13	0.9042 (9)	0.29499 (18)	0.22953 (17)	0.0436 (11)
H13	0.9269	0.2565	0.2072	0.052*
C14	1.0756 (9)	0.31201 (18)	0.26817 (17)	0.0425 (11)
H14	1.2165	0.2851	0.2726	0.051*
C15	1.0426 (9)	0.36864 (16)	0.30087 (15)	0.0371 (9)
H15	1.1612	0.3802	0.3274	0.045*
C16	0.6728 (10)	0.43619 (19)	0.49772 (17)	0.0467 (11)
H16A	0.5836	0.4537	0.5298	0.056*
H16B	0.8329	0.4197	0.5102	0.056*
C17	0.5316 (9)	0.37961 (16)	0.47184 (15)	0.0367 (9)
C18	0.6145 (8)	0.35048 (19)	0.42360 (16)	0.0412 (10)
H18	0.7611	0.3657	0.4073	0.049*
C19	0.4844 (11)	0.29972 (18)	0.39955 (17)	0.0517 (12)
H19	0.5387	0.2812	0.3659	0.062*
C20	0.2765 (11)	0.2752 (2)	0.4234 (2)	0.0604 (15)
H20	0.1886	0.2398	0.4066	0.072*
C21	0.1970 (10)	0.3025 (2)	0.4719 (2)	0.0552 (13)
H21	0.0544	0.2858	0.4889	0.066*
C22	0.3263 (9)	0.35477 (19)	0.49587 (17)	0.0441 (10)
H22	0.2712	0.3734	0.5294	0.053*
C23	0.9028 (8)	0.57286 (17)	0.55147 (15)	0.0371 (10)
H23A	0.9214	0.616	0.5711	0.045*
H23B	0.7359	0.5562	0.5577	0.045*
C24	1.0720 (10)	0.5265 (2)	0.62974 (16)	0.0533 (12)
H24A	0.9042	0.5199	0.6432	0.064*
H24B	1.1306	0.5705	0.643	0.064*
C25	1.2327 (9)	0.4717 (2)	0.65142 (16)	0.0442 (11)
C26	1.1766 (9)	0.4043 (2)	0.64257 (18)	0.0504 (11)
H26	1.0354	0.3925	0.6225	0.06*
C27	1.3272 (9)	0.3549 (2)	0.66308 (18)	0.0511 (11)
H27	1.288	0.309	0.6572	0.061*
C28	1.5319 (10)	0.37129 (19)	0.69168 (16)	0.0496 (11)
H28	1.635	0.3371	0.7056	0.06*

C29	1.5872 (10)	0.4378 (2)	0.70016 (17)	0.0524 (12)
H29	1.7305	0.4496	0.7195	0.063*
C30	1.4362 (9)	0.48721 (18)	0.68078 (17)	0.0470 (10)
H30	1.4737	0.5329	0.6879	0.056*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0362 (2)	0.02898 (18)	0.0534 (2)	0.00229 (17)	−0.00030 (19)	0.00481 (16)
O1	0.0390 (18)	0.0234 (12)	0.0397 (14)	0.0005 (11)	−0.0060 (13)	−0.0011 (10)
O2	0.0385 (17)	0.0174 (10)	0.0419 (12)	0.0030 (11)	−0.0051 (13)	−0.0026 (9)
O3	0.0414 (19)	0.0239 (12)	0.0546 (16)	−0.0036 (12)	−0.0056 (14)	−0.0026 (11)
O4	0.0393 (17)	0.0219 (10)	0.0442 (12)	0.0038 (12)	−0.0062 (14)	−0.0071 (9)
O5	0.0436 (19)	0.0251 (12)	0.0488 (15)	−0.0033 (12)	−0.0060 (13)	0.0063 (11)
O6	0.050 (2)	0.0314 (13)	0.0374 (13)	0.0091 (12)	0.0003 (13)	0.0005 (10)
C1	0.031 (2)	0.0222 (16)	0.041 (2)	0.0025 (15)	−0.0038 (17)	−0.0011 (14)
C2	0.036 (2)	0.0192 (16)	0.044 (2)	0.0052 (15)	−0.0054 (18)	−0.0032 (14)
C3	0.029 (2)	0.0164 (13)	0.0433 (18)	0.0013 (16)	−0.0066 (19)	−0.0051 (12)
C4	0.030 (2)	0.0226 (16)	0.0433 (19)	0.0013 (15)	−0.0044 (17)	−0.0017 (14)
C5	0.035 (3)	0.0235 (16)	0.0432 (19)	0.0036 (16)	−0.0019 (18)	0.0001 (14)
C6	0.034 (3)	0.0189 (15)	0.0402 (19)	−0.0019 (15)	0.0061 (18)	−0.0009 (14)
C7	0.042 (3)	0.0223 (17)	0.045 (2)	0.0051 (16)	0.0039 (19)	−0.0017 (15)
C8	0.045 (3)	0.0234 (17)	0.044 (2)	0.0029 (17)	−0.0037 (19)	−0.0003 (15)
C9	0.040 (2)	0.0240 (17)	0.043 (2)	0.0009 (16)	−0.0043 (18)	−0.0046 (15)
C10	0.035 (2)	0.0242 (17)	0.0384 (19)	−0.0051 (16)	−0.0012 (18)	0.0012 (15)
C11	0.044 (3)	0.0278 (18)	0.044 (2)	−0.0050 (17)	0.000 (2)	−0.0003 (16)
C12	0.053 (3)	0.036 (2)	0.044 (2)	−0.011 (2)	−0.005 (2)	−0.0057 (17)
C13	0.056 (3)	0.0254 (18)	0.049 (2)	−0.0059 (18)	0.005 (2)	−0.0069 (16)
C14	0.043 (3)	0.0265 (18)	0.058 (2)	−0.0018 (17)	0.003 (2)	−0.0060 (17)
C15	0.039 (3)	0.0280 (16)	0.0443 (19)	−0.0057 (17)	−0.0024 (19)	−0.0044 (14)
C16	0.058 (3)	0.033 (2)	0.049 (2)	−0.010 (2)	−0.005 (2)	0.0031 (18)
C17	0.039 (3)	0.0245 (16)	0.047 (2)	0.0043 (18)	−0.002 (2)	0.0058 (14)
C18	0.040 (3)	0.034 (2)	0.049 (2)	0.0060 (19)	−0.0005 (18)	0.0030 (18)
C19	0.068 (4)	0.0322 (19)	0.055 (2)	0.012 (2)	−0.008 (3)	−0.0039 (17)
C20	0.082 (4)	0.027 (2)	0.072 (3)	−0.007 (2)	−0.031 (3)	0.007 (2)
C21	0.045 (3)	0.042 (2)	0.078 (3)	−0.011 (2)	−0.006 (3)	0.021 (2)
C22	0.047 (3)	0.035 (2)	0.050 (2)	0.0075 (19)	0.004 (2)	0.0066 (18)
C23	0.040 (3)	0.0254 (17)	0.046 (2)	0.0060 (16)	−0.0008 (18)	0.0004 (15)
C24	0.057 (4)	0.060 (3)	0.042 (2)	0.015 (2)	0.001 (2)	0.0043 (19)
C25	0.055 (3)	0.041 (2)	0.036 (2)	0.011 (2)	0.011 (2)	0.0016 (17)
C26	0.045 (3)	0.055 (3)	0.051 (2)	0.004 (2)	−0.006 (2)	−0.009 (2)
C27	0.054 (3)	0.034 (2)	0.065 (3)	−0.001 (2)	0.002 (2)	−0.0014 (19)
C28	0.062 (3)	0.0396 (19)	0.047 (2)	0.010 (2)	0.003 (2)	0.0092 (16)
C29	0.062 (4)	0.047 (2)	0.049 (2)	0.000 (2)	−0.011 (2)	0.0013 (19)
C30	0.062 (3)	0.0329 (18)	0.046 (2)	0.0002 (19)	0.000 (3)	0.0022 (18)

Geometric parameters (Å, º)

Br1—C8	1.947 (4)	C12—C13	1.381 (6)
O1—C1	1.397 (4)	C13—H13	0.95
O1—C5	1.428 (5)	C13—C14	1.378 (6)
O2—C1	1.448 (4)	C14—H14	0.95
O2—C6	1.377 (4)	C14—C15	1.395 (5)
O3—C6	1.192 (5)	C15—H15	0.95
O4—C3	1.435 (3)	C16—H16A	0.99
O4—C9	1.407 (5)	C16—H16B	0.99
O5—C4	1.415 (5)	C16—C17	1.510 (6)
O5—C16	1.435 (5)	C17—C18	1.392 (6)
O6—C23	1.423 (5)	C17—C22	1.367 (6)
O6—C24	1.414 (5)	C18—H18	0.95
C1—H1	1	C18—C19	1.373 (6)
C1—C2	1.527 (5)	C19—H19	0.95
C2—H2	1	C19—C20	1.375 (8)
C2—C3	1.528 (6)	C20—H20	0.95
C2—C8	1.523 (5)	C20—C21	1.375 (7)
C3—H3	1	C21—H21	0.95
C3—C4	1.517 (5)	C21—C22	1.392 (6)
C4—H4	1	C22—H22	0.95
C4—C5	1.538 (5)	C23—H23A	0.99
C5—H5	1	C23—H23B	0.99
C5—C23	1.514 (5)	C24—H24A	0.99
C6—C7	1.486 (5)	C24—H24B	0.99
C7—H7A	0.98	C24—C25	1.503 (6)
C7—H7B	0.98	C25—C26	1.394 (6)
C7—H7C	0.98	C25—C30	1.367 (7)
C8—H8A	0.99	C26—H26	0.95
C8—H8B	0.99	C26—C27	1.383 (7)
C9—H9A	0.99	C27—H27	0.95
C9—H9B	0.99	C27—C28	1.367 (7)
C9—C10	1.520 (5)	C28—H28	0.95
C10—C11	1.381 (6)	C28—C29	1.377 (6)
C10—C15	1.381 (6)	C29—H29	0.95
C11—H11	0.95	C29—C30	1.373 (6)
C11—C12	1.399 (5)	C30—H30	0.95
C12—H12	0.95		
C1—O1—C5	114.4 (3)	C13—C12—H12	119.8
C6—O2—C1	115.3 (3)	C12—C13—H13	120.2
C9—O4—C3	114.3 (3)	C14—C13—C12	119.7 (4)
C4—O5—C16	116.9 (3)	C14—C13—H13	120.2
C24—O6—C23	109.8 (3)	C13—C14—H14	119.9
O1—C1—O2	110.3 (3)	C13—C14—C15	120.2 (4)
O1—C1—H1	109.2	C15—C14—H14	119.9
O1—C1—C2	111.8 (3)	C10—C15—C14	120.1 (4)

O2—C1—H1	109.2	C10—C15—H15	119.9
O2—C1—C2	107.0 (3)	C14—C15—H15	119.9
C2—C1—H1	109.2	O5—C16—H16A	109.9
C1—C2—H2	108.1	O5—C16—H16B	109.9
C1—C2—C3	110.4 (3)	O5—C16—C17	109.0 (3)
C3—C2—H2	108.1	H16A—C16—H16B	108.3
C8—C2—C1	113.1 (3)	C17—C16—H16A	109.9
C8—C2—H2	108.1	C17—C16—H16B	109.9
C8—C2—C3	108.9 (3)	C18—C17—C16	119.9 (4)
O4—C3—C2	108.1 (3)	C22—C17—C16	121.1 (4)
O4—C3—H3	108.8	C22—C17—C18	119.0 (4)
O4—C3—C4	110.3 (2)	C17—C18—H18	120
C2—C3—H3	108.8	C19—C18—C17	119.9 (4)
C4—C3—C2	112.0 (3)	C19—C18—H18	120
C4—C3—H3	108.8	C18—C19—H19	119.5
O5—C4—C3	107.5 (3)	C18—C19—C20	121.0 (4)
O5—C4—H4	110.1	C20—C19—H19	119.5
O5—C4—C5	111.4 (3)	C19—C20—H20	120.3
C3—C4—H4	110.1	C19—C20—C21	119.4 (4)
C3—C4—C5	107.7 (3)	C21—C20—H20	120.3
C5—C4—H4	110.1	C20—C21—H21	120.1
O1—C5—C4	109.7 (3)	C20—C21—C22	119.8 (5)
O1—C5—H5	108.3	C22—C21—H21	120.1
O1—C5—C23	107.3 (3)	C17—C22—C21	120.8 (4)
C4—C5—H5	108.3	C17—C22—H22	119.6
C23—C5—C4	114.8 (3)	C21—C22—H22	119.6
C23—C5—H5	108.3	O6—C23—C5	109.9 (3)
O2—C6—C7	109.8 (3)	O6—C23—H23A	109.7
O3—C6—O2	123.1 (3)	O6—C23—H23B	109.7
O3—C6—C7	127.1 (3)	C5—C23—H23A	109.7
C6—C7—H7A	109.5	C5—C23—H23B	109.7
C6—C7—H7B	109.5	H23A—C23—H23B	108.2
C6—C7—H7C	109.5	O6—C24—H24A	109.7
H7A—C7—H7B	109.5	O6—C24—H24B	109.7
H7A—C7—H7C	109.5	O6—C24—C25	109.8 (3)
H7B—C7—H7C	109.5	H24A—C24—H24B	108.2
Br1—C8—H8A	108.8	C25—C24—H24A	109.7
Br1—C8—H8B	108.8	C25—C24—H24B	109.7
C2—C8—Br1	113.6 (3)	C26—C25—C24	121.0 (5)
C2—C8—H8A	108.8	C30—C25—C24	120.3 (4)
C2—C8—H8B	108.8	C30—C25—C26	118.8 (4)
H8A—C8—H8B	107.7	C25—C26—H26	120.1
O4—C9—H9A	109.8	C27—C26—C25	119.8 (5)
O4—C9—H9B	109.8	C27—C26—H26	120.1
O4—C9—C10	109.6 (3)	C26—C27—H27	119.7
H9A—C9—H9B	108.2	C28—C27—C26	120.6 (4)
C10—C9—H9A	109.8	C28—C27—H27	119.7
C10—C9—H9B	109.8	C27—C28—H28	120.3

C11—C10—C9	118.8 (4)	C27—C28—C29	119.4 (4)
C15—C10—C9	121.3 (3)	C29—C28—H28	120.3
C15—C10—C11	119.9 (3)	C28—C29—H29	119.8
C10—C11—H11	120.1	C30—C29—C28	120.3 (5)
C10—C11—C12	119.7 (4)	C30—C29—H29	119.8
C12—C11—H11	120.1	C25—C30—C29	121.0 (4)
C11—C12—H12	119.8	C25—C30—H30	119.5
C13—C12—C11	120.4 (4)	C29—C30—H30	119.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C18—H18···O4	0.95	2.85	3.663 (7)	144