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3,28-Diacetoxy-29-bromobetulin

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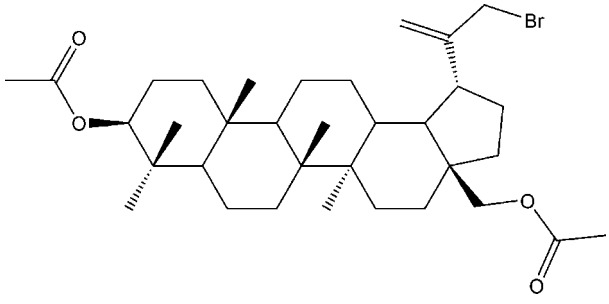
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.006 Å; R factor = 0.057; wR factor = 0.145; data-to-parameter ratio = 15.6.

In the title molecule, $C_{34}H_{53}BrO_4$, all the cyclohexane rings adopt chair conformations, while the cyclopentane ring adopts an envelope conformation. In the crystal, weak intermolecular C—H···O hydrogen bonds link the molecules into corrugated sheets parallel to the *ab* plane.

Related literature

For the anti-HIV and antitumor activities of betulin derivatives, see: Sun *et al.* (1998) and Kim *et al.* (1998), respectively. For a related structure, see Mohamed *et al.* (2006).



Experimental

Crystal data

$C_{34}H_{53}BrO_4$
M_r = 605.67
Orthorhombic, $P2_12_12_1$
a = 7.152 (3) Å
b = 14.946 (7) Å
c = 29.837 (12) Å
V = 3189 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.32 mm^{-1}
T = 291 K
0.40 × 0.38 × 0.37 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.622, T_{max} = 0.642
23319 measured reflections
5578 independent reflections
3534 reflections with $I > 2\sigma(I)$
R_{int} = 0.102

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.145
S = 0.94
5578 reflections
358 parameters
18 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}
Absolute structure: Flack (1983), 2346 Friedel pairs
Flack parameter: 0.024 (12)

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

D—H···A	D—H	H···A	D···A	D—H···A
C32—H32A···O4^{i}	0.96	2.48	3.365 (7)	154
C28—H28B···O4^{ii}	0.97	2.57	3.487 (6)	158

Symmetry codes: (i) $x+1, y-1, z$; (ii) $x, y-1, z$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2584).

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supporting information

Acta Cryst. (2009). E**65**, o1982 [doi:10.1107/S1600536809027640]

3,28-Diacetoxy-29-bromobetulin

Wei-Min Ding, Li-Jia Jing, Tao Yu, Yang Wang and Xiu-Feng Yan

S1. Comment

Betulin and its derivatives have been attracting extensive interests, owing to their anti-HIV and antitumor activites (Sun *et al.*, 1998; Kim *et al.*, 1998). The crystal structure of the diacetylation of betulin has been reported, considering the significance of its stereochemistry study. (Mohamed *et al.*, 2006). We report here the synthesis and the crystal structure of the title compound (I) - a new betulin derivative.

In (I) (Fig. 1), the cyclopentane ring adopts a twisted envelope conformation and all cyclohexane rings adopt chair conformations. The bond distances and angles are all within the expected ranges and agree with those in the similar compound reported previously (Mohamed *et al.*, 2006).

In the crystal, weak intermolecular C—H···O hydrogen bonds (Table 1) link the molecules into corrugated sheets parallel to *ab* plane.

S2. Experimental

Purified betulin (4.4 g, 10 mmol) was dissolved in dichloromethane (100 ml) and pyridine (0.5 ml) mixed solvent, followed by the addition of acetic anhydride (5 ml, 5.3 mmol). The reaction mixture was stirred for 24 h at room temperature. The solvent was removed by distillation under vacuum. The crude product was washed with a small quantity of benzene and then recrystallized from benzene (30 ml), 3.1 g diacetate-betulin was obtained.

The 3, 28-diacetate-betulin (1.0 g, 2 mmol), *N*-bromosuccinimide (0.35 g, 2 mmol) and benzoyl peroxide (0.05 g, 0.22 mmol) were dissolved in tetrachloromethane (50 ml). The reaction mixture was stirred for 6 h at reflux temperature. The solvent was removed by distillation under vacuum. The crude product was washed with a small quantity of ethanol and then recrystallized from petroleum ether (6 ml), 0.4 g suitable for X-ray diffraction test colourless block crystals of the titel compound was obtained.

S3. Refinement

C-bound H-atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.96-0.98 Å and U_{iso}(H) = 1.2-1.5Ueq(C).

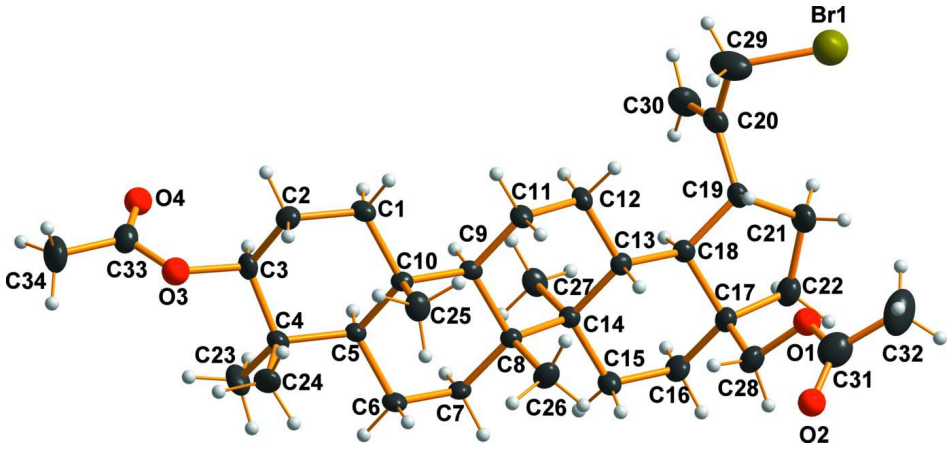

Figure 1

The molecular structure of (I) showing the atomic numbering and 50% probability displacement ellipsoids.

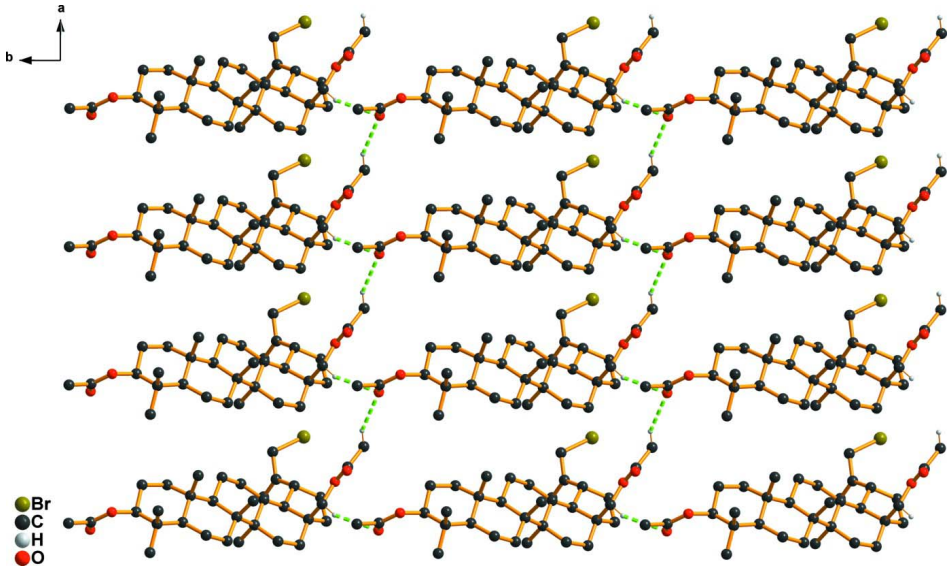

Figure 2

A portion of the crystal, showing the two-dimensional sheet of hydrogen-bonded (dashed lines) molecules. H atoms not involved in hydrogen bonds have been omitted.

3,28-Diacetoxy-29-bromobetulin

Crystal data

$C_{34}H_{53}BrO_4$
$M_r = 605.67$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 7.152\ (3)$ Å
$b = 14.946\ (7)$ Å
$c = 29.837\ (12)$ Å
$V = 3189\ (2)$ Å^3
$Z = 4$

$F(000) = 1296$
$D_x = 1.261$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 15343 reflections
$\theta = 3.1–27.4°$
$\mu = 1.32$ mm^{-1}
$T = 291$ K
Block, colourless
0.40 × 0.38 × 0.37 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.622, T_{max} = 0.642
23319 measured reflections
5578 independent reflections
3534 reflections with $I > 2\sigma(I)$
R_{int} = 0.102
θ_{max} = 25.0°, θ_{min} = 3.1°
h = −8→8
k = −17→17
l = −35→35

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.145
S = 0.94
5578 reflections
358 parameters
18 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
$w = 1/[\sigma^2(F_o^2) + (0.066P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}
Absolute structure: Flack (1983), 2346 Friedel pairs
Absolute structure parameter: 0.024 (12)

Special details

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	1.44746 (8)	−0.01367 (6)	0.18762 (2)	0.1130 (4)
C1	1.0979 (5)	0.4597 (3)	0.38899 (13)	0.0394 (10)
H1A	1.2251	0.4468	0.3798	0.047*
H1B	1.0239	0.4685	0.3621	0.047*
C2	1.0967 (5)	0.5466 (3)	0.41652 (14)	0.0419 (10)
H2A	1.1780	0.5401	0.4423	0.050*
H2B	1.1437	0.5955	0.3984	0.050*
C3	0.9011 (6)	0.5672 (3)	0.43183 (13)	0.0384 (10)
H3	0.8215	0.5744	0.4053	0.046*
C4	0.8156 (5)	0.4940 (3)	0.46193 (12)	0.0366 (9)
C5	0.8272 (5)	0.4054 (3)	0.43421 (12)	0.0311 (9)
H5	0.7495	0.4169	0.4078	0.037*
C6	0.7341 (6)	0.3239 (3)	0.45702 (14)	0.0430 (11)
H6A	0.8162	0.3006	0.4801	0.052*
H6B	0.6182	0.3424	0.4712	0.052*

C7	0.6935 (5)	0.2506 (3)	0.42254 (13)	0.0380 (10)
H7A	0.6044	0.2733	0.4008	0.046*
H7B	0.6360	0.2002	0.4377	0.046*
C8	0.8677 (5)	0.2177 (3)	0.39760 (12)	0.0308 (9)
C9	0.9820 (4)	0.3005 (3)	0.37983 (11)	0.0294 (8)
H9	0.9009	0.3276	0.3571	0.035*
C10	1.0203 (4)	0.3785 (3)	0.41474 (11)	0.0281 (8)
C11	1.1536 (5)	0.2692 (3)	0.35383 (13)	0.0379 (10)
H11A	1.2395	0.2404	0.3744	0.045*
H11B	1.2164	0.3211	0.3413	0.045*
C12	1.1069 (5)	0.2039 (3)	0.31566 (13)	0.0367 (9)
H12A	1.0373	0.2354	0.2926	0.044*
H12B	1.2223	0.1825	0.3024	0.044*
C13	0.9924 (5)	0.1240 (3)	0.33177 (12)	0.0310 (9)
H13	1.0681	0.0934	0.3544	0.037*
C14	0.8095 (5)	0.1562 (3)	0.35600 (12)	0.0324 (9)
C15	0.6922 (6)	0.0744 (3)	0.37207 (14)	0.0445 (11)
H15A	0.7519	0.0492	0.3984	0.053*
H15B	0.5695	0.0955	0.3810	0.053*
C16	0.6672 (6)	−0.0005 (3)	0.33708 (15)	0.0503 (11)
H16A	0.6061	−0.0512	0.3511	0.060*
H16B	0.5865	0.0208	0.3132	0.060*
C17	0.8514 (6)	−0.0310 (3)	0.31707 (13)	0.0423 (10)
C18	0.9435 (5)	0.0536 (3)	0.29593 (12)	0.0341 (9)
H18	0.8472	0.0810	0.2770	0.041*
C19	1.0936 (5)	0.0189 (3)	0.26382 (12)	0.0395 (9)
H19	1.2080	0.0073	0.2809	0.047*
C20	1.1404 (6)	0.0818 (3)	0.22529 (15)	0.0500 (12)
C21	1.0121 (7)	−0.0725 (3)	0.24745 (15)	0.0582 (13)
H21A	0.9779	−0.0688	0.2160	0.070*
H21B	1.1044	−0.1195	0.2509	0.070*
C22	0.8384 (7)	−0.0930 (3)	0.27615 (16)	0.0549 (12)
H22A	0.7249	−0.0811	0.2594	0.066*
H22B	0.8381	−0.1552	0.2854	0.066*
C23	0.6078 (6)	0.5185 (3)	0.46879 (16)	0.0609 (14)
H23A	0.5525	0.4779	0.4899	0.091*
H23B	0.5431	0.5144	0.4407	0.091*
H23C	0.5987	0.5785	0.4801	0.091*
C24	0.9094 (6)	0.4905 (3)	0.50812 (12)	0.0520 (11)
H24A	1.0428	0.4891	0.5045	0.078*
H24B	0.8693	0.4377	0.5237	0.078*
H24C	0.8749	0.5426	0.5251	0.078*
C25	1.1663 (5)	0.3518 (3)	0.45069 (14)	0.0448 (11)
H25A	1.2627	0.3163	0.4371	0.067*
H25B	1.1063	0.3177	0.4739	0.067*
H25C	1.2205	0.4048	0.4634	0.067*
C26	0.9830 (6)	0.1609 (3)	0.43158 (12)	0.0464 (11)
H26A	0.9780	0.1885	0.4606	0.070*

H26B	1.1106	0.1575	0.4218	0.070*
H26C	0.9314	0.1017	0.4333	0.070*
C27	0.6862 (5)	0.2109 (3)	0.32242 (13)	0.0416 (10)
H27A	0.6494	0.1730	0.2980	0.062*
H27B	0.7568	0.2606	0.3111	0.062*
H27C	0.5768	0.2325	0.3376	0.062*
C28	0.9630 (7)	−0.0780 (3)	0.35488 (15)	0.0532 (12)
H28A	1.0021	−0.0341	0.3769	0.064*
H28B	0.8833	−0.1213	0.3698	0.064*
C29	1.3386 (8)	0.0938 (4)	0.21333 (19)	0.0841 (19)
H29A	1.4084	0.1103	0.2399	0.101*
H29B	1.3494	0.1424	0.1919	0.101*
C30	1.0135 (8)	0.1247 (4)	0.20139 (15)	0.0696 (16)
H30A	1.0504	0.1613	0.1778	0.084*
H30B	0.8873	0.1184	0.2082	0.084*
C31	1.2306 (8)	−0.1665 (4)	0.3652 (2)	0.0732 (16)
C32	1.3789 (7)	−0.2221 (4)	0.3428 (2)	0.094 (2)
H32A	1.4944	−0.2161	0.3589	0.141*
H32B	1.3957	−0.2019	0.3126	0.141*
H32C	1.3411	−0.2837	0.3427	0.141*
C33	0.8263 (7)	0.7245 (3)	0.43739 (18)	0.0534 (12)
C34	0.8185 (9)	0.8011 (3)	0.46859 (19)	0.0819 (18)
H34A	0.9413	0.8261	0.4719	0.123*
H34B	0.7738	0.7812	0.4972	0.123*
H34C	0.7354	0.8458	0.4569	0.123*
O1	1.1239 (5)	−0.1224 (2)	0.33705 (11)	0.0625 (9)
O2	1.2042 (7)	−0.1640 (4)	0.40533 (18)	0.1280 (19)
O3	0.8996 (4)	0.65128 (19)	0.45704 (10)	0.0473 (7)
O4	0.7726 (6)	0.7259 (2)	0.39882 (13)	0.0783 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0718 (4)	0.1855 (9)	0.0816 (4)	0.0435 (5)	0.0114 (3)	−0.0252 (5)
C1	0.039 (2)	0.044 (3)	0.035 (2)	−0.0083 (19)	0.0101 (17)	−0.0059 (19)
C2	0.047 (2)	0.037 (3)	0.042 (2)	−0.010 (2)	0.0035 (19)	−0.0031 (19)
C3	0.050 (2)	0.032 (2)	0.033 (2)	0.0026 (19)	−0.0026 (18)	−0.0114 (18)
C4	0.0410 (19)	0.038 (2)	0.0307 (19)	0.001 (2)	0.0084 (16)	−0.0040 (19)
C5	0.0329 (19)	0.032 (2)	0.029 (2)	−0.0004 (17)	0.0074 (16)	0.0006 (17)
C6	0.046 (2)	0.042 (3)	0.041 (2)	−0.008 (2)	0.0186 (19)	−0.001 (2)
C7	0.044 (2)	0.033 (2)	0.038 (2)	−0.0085 (19)	0.0154 (18)	0.0018 (19)
C8	0.0327 (18)	0.031 (2)	0.0285 (19)	−0.0018 (17)	0.0032 (16)	0.0014 (17)
C9	0.0260 (17)	0.036 (2)	0.0257 (18)	0.0005 (16)	0.0028 (14)	0.0018 (16)
C10	0.0263 (18)	0.033 (2)	0.0250 (18)	−0.0032 (16)	−0.0006 (15)	0.0006 (17)
C11	0.0321 (19)	0.038 (2)	0.044 (2)	−0.0041 (19)	0.0077 (17)	−0.0048 (19)
C12	0.0346 (19)	0.034 (2)	0.041 (2)	−0.0028 (17)	0.0098 (18)	−0.0103 (19)
C13	0.0304 (18)	0.031 (2)	0.032 (2)	0.0034 (16)	−0.0028 (15)	0.0021 (17)
C14	0.0334 (18)	0.031 (2)	0.032 (2)	−0.0026 (18)	0.0030 (16)	−0.0023 (17)

C15	0.046 (2)	0.039 (3)	0.048 (3)	−0.005 (2)	0.013 (2)	−0.006 (2)
C16	0.056 (2)	0.037 (3)	0.058 (3)	−0.016 (2)	0.008 (2)	−0.008 (2)
C17	0.055 (2)	0.031 (2)	0.041 (2)	−0.002 (2)	0.0046 (19)	−0.004 (2)
C18	0.0378 (18)	0.034 (2)	0.0303 (18)	0.0008 (18)	−0.0003 (16)	−0.0001 (17)
C19	0.050 (2)	0.037 (2)	0.0312 (19)	0.004 (2)	0.0009 (16)	−0.0066 (18)
C20	0.060 (3)	0.049 (3)	0.041 (2)	−0.003 (2)	0.010 (2)	−0.012 (2)
C21	0.083 (3)	0.045 (3)	0.047 (3)	−0.004 (3)	0.009 (2)	−0.012 (2)
C22	0.069 (3)	0.038 (3)	0.058 (3)	−0.007 (2)	0.001 (2)	−0.007 (2)
C23	0.049 (2)	0.052 (3)	0.082 (3)	−0.001 (2)	0.024 (2)	−0.021 (3)
C24	0.078 (3)	0.049 (3)	0.029 (2)	−0.007 (3)	0.0065 (19)	−0.004 (2)
C25	0.042 (2)	0.047 (3)	0.045 (2)	0.006 (2)	−0.0109 (19)	−0.005 (2)
C26	0.066 (3)	0.041 (3)	0.033 (2)	0.001 (2)	−0.008 (2)	0.007 (2)
C27	0.0335 (19)	0.051 (3)	0.041 (2)	0.0097 (19)	−0.0099 (17)	−0.004 (2)
C28	0.072 (3)	0.036 (3)	0.052 (3)	0.003 (3)	0.003 (2)	0.007 (2)
C29	0.075 (3)	0.115 (5)	0.062 (3)	−0.033 (4)	0.011 (3)	−0.009 (4)
C30	0.089 (4)	0.078 (4)	0.041 (3)	0.016 (3)	0.004 (3)	0.008 (3)
C31	0.066 (3)	0.074 (4)	0.079 (4)	−0.009 (3)	−0.014 (3)	0.001 (4)
C32	0.065 (3)	0.064 (4)	0.153 (6)	0.014 (3)	−0.022 (4)	−0.005 (4)
C33	0.056 (3)	0.040 (3)	0.064 (3)	−0.004 (2)	0.006 (2)	−0.006 (3)
C34	0.107 (4)	0.046 (3)	0.093 (4)	−0.001 (3)	0.018 (4)	−0.025 (3)
O1	0.078 (2)	0.054 (2)	0.055 (2)	0.0185 (19)	−0.0040 (17)	0.0052 (17)
O2	0.109 (3)	0.179 (5)	0.097 (3)	0.039 (3)	−0.026 (3)	0.022 (3)
O3	0.0630 (18)	0.0353 (18)	0.0435 (16)	0.0022 (15)	−0.0035 (14)	−0.0104 (14)
O4	0.108 (3)	0.049 (2)	0.078 (3)	0.013 (2)	−0.027 (2)	0.005 (2)

Geometric parameters (Å, º)

Br1—C29	1.943 (6)	C17—C28	1.550 (6)
C1—C2	1.537 (5)	C17—C18	1.559 (5)
C1—C10	1.539 (5)	C18—C19	1.530 (5)
C1—H1A	0.9700	C18—H18	0.9800
C1—H1B	0.9700	C19—C20	1.522 (6)
C2—C3	1.504 (6)	C19—C21	1.563 (6)
C2—H2A	0.9700	C19—H19	0.9800
C2—H2B	0.9700	C20—C30	1.320 (6)
C3—O3	1.464 (5)	C20—C29	1.473 (7)
C3—C4	1.542 (6)	C21—C22	1.540 (7)
C3—H3	0.9800	C21—H21A	0.9700
C4—C24	1.534 (5)	C21—H21B	0.9700
C4—C23	1.544 (5)	C22—H22A	0.9700
C4—C5	1.563 (5)	C22—H22B	0.9700
C5—C6	1.546 (5)	C23—H23A	0.9600
C5—C10	1.551 (5)	C23—H23B	0.9600
C5—H5	0.9800	C23—H23C	0.9600
C6—C7	1.531 (6)	C24—H24A	0.9600
C6—H6A	0.9700	C24—H24B	0.9600
C6—H6B	0.9700	C24—H24C	0.9600
C7—C8	1.532 (5)	C25—H25A	0.9600

C7—H7A	0.9700	C25—H25B	0.9600
C7—H7B	0.9700	C25—H25C	0.9600
C8—C26	1.559 (5)	C26—H26A	0.9600
C8—C9	1.574 (5)	C26—H26B	0.9600
C8—C14	1.600 (5)	C26—H26C	0.9600
C9—C11	1.525 (5)	C27—H27A	0.9600
C9—C10	1.588 (5)	C27—H27B	0.9600
C9—H9	0.9800	C27—H27C	0.9600
C10—C25	1.550 (5)	C28—O1	1.431 (5)
C11—C12	1.537 (5)	C28—H28A	0.9700
C11—H11A	0.9700	C28—H28B	0.9700
C11—H11B	0.9700	C29—H29A	0.9700
C12—C13	1.526 (5)	C29—H29B	0.9700
C12—H12A	0.9700	C30—H30A	0.9300
C12—H12B	0.9700	C30—H30B	0.9300
C13—C18	1.540 (5)	C31—O2	1.211 (7)
C13—C14	1.570 (5)	C31—O1	1.313 (6)
C13—H13	0.9800	C31—C32	1.504 (8)
C14—C15	1.558 (5)	C32—H32A	0.9600
C14—C27	1.565 (5)	C32—H32B	0.9600
C15—C16	1.541 (5)	C32—H32C	0.9600
C15—H15A	0.9700	C33—O4	1.213 (6)
C15—H15B	0.9700	C33—O3	1.348 (6)
C16—C17	1.516 (6)	C33—C34	1.477 (7)
C16—H16A	0.9700	C34—H34A	0.9600
C16—H16B	0.9700	C34—H34B	0.9600
C17—C22	1.536 (6)	C34—H34C	0.9600

C2—C1—C10	113.4 (3)	C16—C17—C28	107.3 (4)
C2—C1—H1A	108.9	C22—C17—C28	109.6 (4)
C10—C1—H1A	108.9	C16—C17—C18	106.4 (3)
C2—C1—H1B	108.9	C22—C17—C18	101.1 (3)
C10—C1—H1B	108.9	C28—C17—C18	116.4 (3)
H1A—C1—H1B	107.7	C19—C18—C13	120.5 (3)
C3—C2—C1	109.9 (3)	C19—C18—C17	106.0 (3)
C3—C2—H2A	109.7	C13—C18—C17	111.6 (3)
C1—C2—H2A	109.7	C19—C18—H18	105.9
C3—C2—H2B	109.7	C13—C18—H18	105.9
C1—C2—H2B	109.7	C17—C18—H18	105.9
H2A—C2—H2B	108.2	C20—C19—C18	114.7 (3)
O3—C3—C2	109.8 (3)	C20—C19—C21	112.7 (3)
O3—C3—C4	107.9 (3)	C18—C19—C21	103.3 (3)
C2—C3—C4	113.6 (3)	C20—C19—H19	108.6
O3—C3—H3	108.5	C18—C19—H19	108.6
C2—C3—H3	108.5	C21—C19—H19	108.6
C4—C3—H3	108.5	C30—C20—C29	118.1 (5)
C24—C4—C3	112.0 (3)	C30—C20—C19	123.9 (4)
C24—C4—C23	108.1 (3)	C29—C20—C19	118.0 (5)

C3—C4—C23	106.9 (3)	C22—C21—C19	107.5 (4)
C24—C4—C5	115.1 (3)	C22—C21—H21A	110.2
C3—C4—C5	105.8 (3)	C19—C21—H21A	110.2
C23—C4—C5	108.8 (3)	C22—C21—H21B	110.2
C6—C5—C10	110.1 (3)	C19—C21—H21B	110.2
C6—C5—C4	114.3 (3)	H21A—C21—H21B	108.5
C10—C5—C4	117.7 (3)	C17—C22—C21	105.9 (4)
C6—C5—H5	104.4	C17—C22—H22A	110.6
C10—C5—H5	104.4	C21—C22—H22A	110.6
C4—C5—H5	104.4	C17—C22—H22B	110.6
C7—C6—C5	110.5 (3)	C21—C22—H22B	110.6
C7—C6—H6A	109.6	H22A—C22—H22B	108.7
C5—C6—H6A	109.6	C4—C23—H23A	109.5
C7—C6—H6B	109.6	C4—C23—H23B	109.5
C5—C6—H6B	109.6	H23A—C23—H23B	109.5
H6A—C6—H6B	108.1	C4—C23—H23C	109.5
C6—C7—C8	113.7 (3)	H23A—C23—H23C	109.5
C6—C7—H7A	108.8	H23B—C23—H23C	109.5
C8—C7—H7A	108.8	C4—C24—H24A	109.5
C6—C7—H7B	108.8	C4—C24—H24B	109.5
C8—C7—H7B	108.8	H24A—C24—H24B	109.5
H7A—C7—H7B	107.7	C4—C24—H24C	109.5
C7—C8—C26	106.8 (3)	H24A—C24—H24C	109.5
C7—C8—C9	109.5 (3)	H24B—C24—H24C	109.5
C26—C8—C9	111.9 (3)	C10—C25—H25A	109.5
C7—C8—C14	110.5 (3)	C10—C25—H25B	109.5
C26—C8—C14	109.2 (3)	H25A—C25—H25B	109.5
C9—C8—C14	109.0 (3)	C10—C25—H25C	109.5
C11—C9—C8	110.4 (3)	H25A—C25—H25C	109.5
C11—C9—C10	114.8 (3)	H25B—C25—H25C	109.5
C8—C9—C10	116.5 (3)	C8—C26—H26A	109.5
C11—C9—H9	104.5	C8—C26—H26B	109.5
C8—C9—H9	104.5	H26A—C26—H26B	109.5
C10—C9—H9	104.5	C8—C26—H26C	109.5
C1—C10—C25	107.8 (3)	H26A—C26—H26C	109.5
C1—C10—C5	107.7 (3)	H26B—C26—H26C	109.5
C25—C10—C5	114.1 (3)	C14—C27—H27A	109.5
C1—C10—C9	108.3 (3)	C14—C27—H27B	109.5
C25—C10—C9	112.4 (3)	H27A—C27—H27B	109.5
C5—C10—C9	106.4 (3)	C14—C27—H27C	109.5
C9—C11—C12	113.4 (3)	H27A—C27—H27C	109.5
C9—C11—H11A	108.9	H27B—C27—H27C	109.5
C12—C11—H11A	108.9	O1—C28—C17	110.7 (3)
C9—C11—H11B	108.9	O1—C28—H28A	109.5
C12—C11—H11B	108.9	C17—C28—H28A	109.5
H11A—C11—H11B	107.7	O1—C28—H28B	109.5
C13—C12—C11	112.4 (3)	C17—C28—H28B	109.5
C13—C12—H12A	109.1	H28A—C28—H28B	108.1

C11—C12—H12A	109.1	C20—C29—Br1	112.4 (4)
C13—C12—H12B	109.1	C20—C29—H29A	109.1
C11—C12—H12B	109.1	Br1—C29—H29A	109.1
H12A—C12—H12B	107.9	C20—C29—H29B	109.1
C12—C13—C18	116.0 (3)	Br1—C29—H29B	109.1
C12—C13—C14	110.6 (3)	H29A—C29—H29B	107.9
C18—C13—C14	109.9 (3)	C20—C30—H30A	120.0
C12—C13—H13	106.6	C20—C30—H30B	120.0
C18—C13—H13	106.6	H30A—C30—H30B	120.0
C14—C13—H13	106.6	O2—C31—O1	121.8 (6)
C15—C14—C27	107.6 (3)	O2—C31—C32	124.4 (6)
C15—C14—C13	110.5 (3)	O1—C31—C32	113.7 (6)
C27—C14—C13	109.6 (3)	C31—C32—H32A	109.5
C15—C14—C8	110.6 (3)	C31—C32—H32B	109.5
C27—C14—C8	110.1 (3)	H32A—C32—H32B	109.5
C13—C14—C8	108.5 (3)	C31—C32—H32C	109.5
C16—C15—C14	115.1 (3)	H32A—C32—H32C	109.5
C16—C15—H15A	108.5	H32B—C32—H32C	109.5
C14—C15—H15A	108.5	O4—C33—O3	123.4 (4)
C16—C15—H15B	108.5	O4—C33—C34	124.9 (5)
C14—C15—H15B	108.5	O3—C33—C34	111.7 (5)
H15A—C15—H15B	107.5	C33—C34—H34A	109.5
C17—C16—C15	112.6 (3)	C33—C34—H34B	109.5
C17—C16—H16A	109.1	H34A—C34—H34B	109.5
C15—C16—H16A	109.1	C33—C34—H34C	109.5
C17—C16—H16B	109.1	H34A—C34—H34C	109.5
C15—C16—H16B	109.1	H34B—C34—H34C	109.5
H16A—C16—H16B	107.8	C31—O1—C28	117.5 (4)
C16—C17—C22	116.2 (3)	C33—O3—C3	118.4 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C32—H32*A*···O4^{i}	0.96	2.48	3.365 (7)	154
C28—H28*B*···O4^{ii}	0.97	2.57	3.487 (6)	158

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