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3,5,7-Triacetoxy-2-(3,4-diacetoxyphenyl)-4*H*-1-benzopyran-4-one

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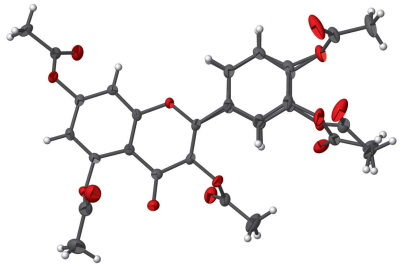
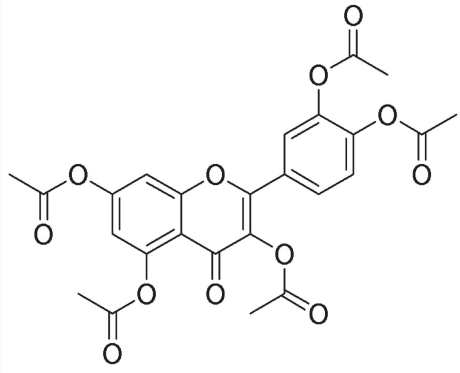
In the title compound, $C_{25}H_{20}O_{12}$, commonly known as pentaacetylated quercetin, the benzene ring and one of its methoxy substituent groups is disordered (site occupancy ratio 0.523:0.427), with a dihedral angle between the major-disorder component and the benzene ring of the benzopyranone moiety of 10.8 (6)°. In the crystal, C—H···O hydrogen-bonding interactions give chains which extend along *b*.

3D view



Chemical scheme



Structure description

Quercetin is the most studied and an important class of flavonoid found in vegetables, fruits and grains. Quercitin and its derivatives are valuable due to their important properties as anti-oxidative (Chopra *et al.*, 2000), anticarcinogenic (Pereira *et al.*, 1996), anti-inflammatory (Ferry *et al.*, 1996) and anti-aggregatory agents (Pignatelli *et al.*, 2000) and their vasodilating effects (Perez-Vizcaino *et al.*, 2002). Acetylated quercetin derivatives have been used as HIV-1 integrase inhibitors for the treatment of HIV-1 infection (Li *et al.*, 2014) as well as to evaluate the cell proliferation inhibition and apoptosis in HL-60 cells (Sakao *et al.*, 2009). Thus, the elucidation of the crystal structures of quercetin derivatives has attracted much attention. Here,we report the crystal structure of the title compound, the pentaacetyl-substituted quercitin derivative, $C_{25}H_{20}O_{12}$.

In the title compound (Fig. 1), the benzene ring and one of its methoxy substituent groups, defined by C1*A*—C14*A*···C2*A*— O1*A*—C6*AA*—O2*A* is disordered, giving the alternative component C1*B*—C14*B*···C2*B*—O1*B*—C6*B*— O2*B* with a site occupancy ratio of 0.523:0.477. The dihedral angle between the major component (*A*) and the benzene ring of the benzopyranone moiety (defined by C2—C3···C12) is 10.8 (6)°. The

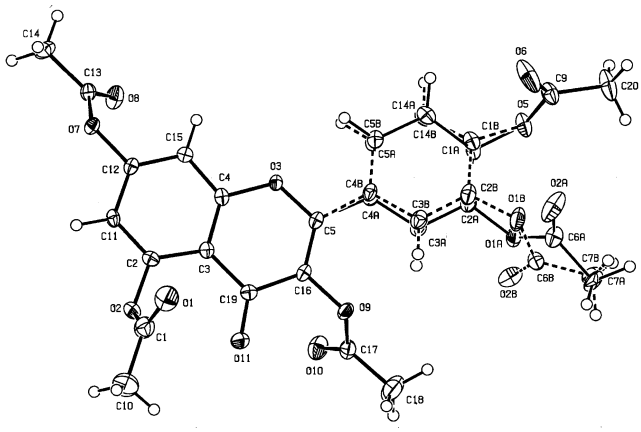


Figure 1
Molecular configuration and atom-numbering scheme for the title compound with displacement ellipsoids drawn at the 40% probability level. The bonds in the minor disordered *B* portion of the molecule are shown as dashed lines.

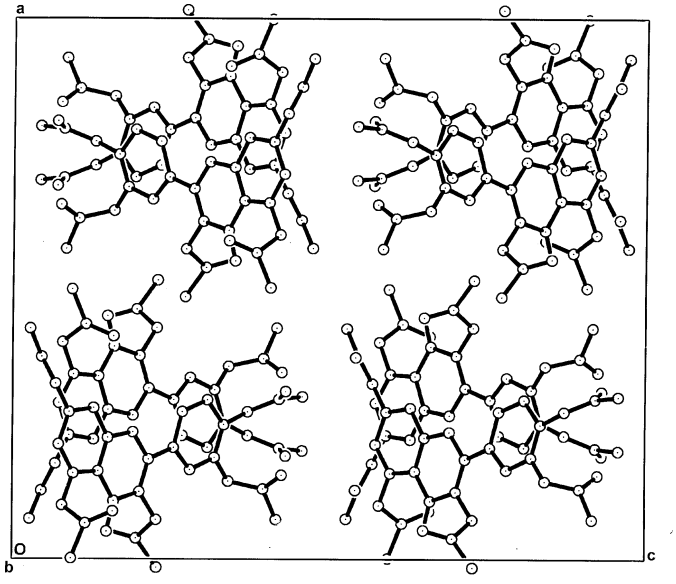


Figure 2
Crystal packing diagram of the title compound, viewed along the *b* axis, with hydrogen atoms and the disordered portion omitted for clarity.

conformations of the two acetyl groups *A* and *B* are very different [torsion angles C1*A*/*B* —C2*A*/*B*—O1*A*/*B*—C6*A*/*B* are 67.6 (12) and −106.1 (11)°, respectively]. In the crystal (Fig. 2), only weak C—H···O hydrogen-bonding interactions are present (Table 1), giving chains extending along *b*.

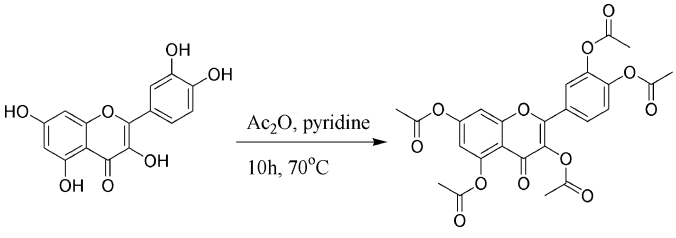


Figure 3
Reaction scheme for the synthesis of the title compound.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7*A*—H7*AB*···O8^{i}	0.96	2.43	3.331 (17)	156
C20—H20*A*···O2*A*^{ii}	0.96	2.33	3.142 (7)	142
C20—H20*B*···O6^{ii}	0.96	2.51	3.352 (5)	146

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

For background and medical applications of quercitin derivatives, see: Chopra *et al.* (2000); Pereira *et al.* (1996); Ferry *et al.* (1996); Pignatelli *et al.* (2000); Perez-Vizcaino *et al.* (2002); Li *et al.* (2014); Sakao *et al.* (2009).

Synthesis and crystallization

The title compound was synthesized as follows (Fig. 3). Acetic anhydride (1 ml) was added to a solution of quercetin (2 mmol) in anhydrous pyridine (8 ml) at room temperature. The reaction mixture was stirred for 10 h at 343 K. After completion of reaction, the resultant mixture was cooled to room temperature, then poured into ice-cold water. The precipitate was separated by filtration and then washed with ice-cold water. The resulting precipitate was filtered and finally recrystallized from methanol. Single crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of an ethanolic solution at room temperature.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{25}H_{20}O_{12}$
M_r	512.41
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	90
a, *b*, *c* (Å)	20.944 (6), 9.316 (3), 24.309 (7)
V (Å^3)	4743 (2)
Z	8
Radiation type	Mo *Kα*
μ (mm^{-1})	0.12
Crystal size (mm)	0.30 × 0.20 × 0.15
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.805, 0.983
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	42484, 4165, 3329
R_{int}	0.065
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.594
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.062, 0.179, 1.21
No. of reflections	4145
No. of parameters	429
No. of restraints	775
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.39

Computer programs: *APEX2* (Bruker, 2009), *SAINT* (Bruker, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008), *PLATON* (Spek, 2009).

m.p. 465–467 K; δ_H (400 MHz $CDCl_3$) 7.75–7.67 (2H, *m*), 7.36 (1H, *s*), 7.33 (1H, *J* = 1.8 Hz, *d*), 6.87 (1H, *J* = 2.1 Hz, *d*), 2.42 (3H, *s*), 2.37–2.31 (12H, *m*). FABMS: MH^+, 511.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Disorder was identified in the benzene ring and one of its acetyl substituent groups and the major and minor components *A* and *B* were included with refined occupancy factors of 0.523 (5) and 0.477 (5), respectively. Two low-angle reflections were considered to be affected by the beamstop during data collection, prompting a *B*-Alert in the *checkCIF* report.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x160028 [doi:10.1107/S2414314616000286]

3,5,7-Triacetoxy-2-(3,4-diacetoxyphenyl)-4*H*-1-benzopyran-4-one

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Crystal data

$C_{25}H_{20}O_{12}$
M_r = 512.41
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
a = 20.944 (6) Å
b = 9.316 (3) Å
c = 24.309 (7) Å
V = 4743 (2) Å^3
Z = 8
F(000) = 2128
D_x = 1.435 Mg m^{-3}
Melting point = 465–467 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 25664 reflections
θ = 1.7–19.9°
μ = 0.12 mm^{-1}
T = 90 K
Prism, yellow
0.30 × 0.20 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.333 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.805, T_{max} = 0.983
42484 measured reflections
4165 independent reflections
3329 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
θ_{max} = 25.0°, θ_{min} = 1.7°
h = −24→24
k = −11→11
l = −28→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.179
S = 1.21
4145 reflections
429 parameters
775 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0743P)^2 + 6.2477P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.06601 (18)	1.1281 (4)	0.11576 (17)	0.0369 (9)	
C2	0.15863 (14)	0.9957 (3)	0.09647 (13)	0.0200 (7)	
C3	0.16239 (14)	0.8854 (3)	0.13579 (12)	0.0177 (6)	
C4	0.22412 (14)	0.8435 (3)	0.15093 (12)	0.0181 (6)	
C5	0.18454 (15)	0.6718 (3)	0.21506 (12)	0.0199 (7)	
C9	0.29542 (18)	0.3080 (4)	0.42104 (14)	0.0307 (8)	
C10	0.0014 (2)	1.1652 (5)	0.0935 (2)	0.0582 (13)	
H10A	−0.0287	1.0927	0.1042	0.087*	
H10B	0.0034	1.1704	0.0541	0.087*	
H10C	−0.0118	1.2564	0.1080	0.087*	
C11	0.21171 (15)	1.0560 (3)	0.07290 (12)	0.0195 (7)	
H11	0.2080	1.1275	0.0464	0.023*	
C12	0.27162 (14)	1.0068 (3)	0.08978 (12)	0.0172 (6)	
C13	0.37350 (15)	1.0017 (4)	0.04557 (13)	0.0226 (7)	
C14	0.42571 (16)	1.1000 (4)	0.02728 (14)	0.0309 (8)	
H14A	0.4663	1.0584	0.0362	0.046*	
H14B	0.4214	1.1908	0.0456	0.046*	
H14C	0.4229	1.1140	−0.0118	0.046*	
C15	0.27911 (14)	0.9016 (3)	0.12845 (12)	0.0178 (6)	
H15	0.3194	0.8703	0.1392	0.021*	
C16	0.12419 (14)	0.7042 (3)	0.20045 (12)	0.0199 (7)	
C17	0.03850 (16)	0.5420 (4)	0.19534 (14)	0.0286 (8)	
C18	−0.0154 (2)	0.4829 (5)	0.22801 (18)	0.0556 (12)	
H18A	−0.0424	0.5600	0.2401	0.083*	
H18B	0.0010	0.4325	0.2594	0.083*	
H18C	−0.0397	0.4179	0.2056	0.083*	
C19	0.10733 (14)	0.8136 (3)	0.16025 (12)	0.0186 (7)	
C20	0.3000 (2)	0.1807 (4)	0.45795 (15)	0.0466 (11)	
H20A	0.3303	0.1138	0.4431	0.070*	
H20B	0.2590	0.1355	0.4608	0.070*	
H20C	0.3138	0.2109	0.4938	0.070*	
C1A	0.2481 (7)	0.371 (2)	0.3341 (8)	0.0212 (19)	0.523 (5)
C2A	0.1875 (3)	0.3714 (7)	0.3196 (3)	0.0214 (13)	0.523 (5)
C3A	0.1651 (3)	0.4643 (8)	0.2808 (3)	0.0210 (14)	0.523 (5)
H3AA	0.1221	0.4626	0.2716	0.025*	0.523 (5)
C4A	0.2058 (14)	0.562 (3)	0.2545 (12)	0.021 (2)	0.523 (5)
C5A	0.2722 (12)	0.5569 (19)	0.2685 (7)	0.021 (2)	0.523 (5)
H5AA	0.3009	0.6171	0.2506	0.025*	0.523 (5)
C6A	0.1296 (3)	0.2842 (7)	0.3962 (3)	0.0299 (15)	0.523 (5)
C7A	0.0733 (8)	0.2110 (15)	0.4167 (7)	0.035 (3)	0.523 (5)

H7AA	0.0580	0.1453	0.3893	0.053*	0.523 (5)
H7AB	0.0840	0.1589	0.4495	0.053*	0.523 (5)
H7AC	0.0407	0.2801	0.4249	0.053*	0.523 (5)
C1B	0.2584 (8)	0.380 (2)	0.3359 (9)	0.021 (2)	0.477 (5)
C2B	0.1922 (4)	0.4237 (9)	0.3362 (3)	0.0271 (15)	0.477 (5)
C3B	0.1683 (4)	0.5189 (9)	0.2988 (3)	0.0247 (16)	0.477 (5)
H3BA	0.1256	0.5462	0.2998	0.030*	0.477 (5)
C4B	0.2102 (16)	0.575 (3)	0.2584 (13)	0.020 (2)	0.477 (5)
C5B	0.2721 (13)	0.538 (2)	0.2600 (8)	0.021 (2)	0.477 (5)
H5BA	0.3003	0.5804	0.2352	0.025*	0.477 (5)
C6B	0.1097 (4)	0.2669 (7)	0.3641 (3)	0.0257 (15)	0.477 (5)
C7B	0.0828 (9)	0.1796 (16)	0.4078 (7)	0.034 (4)	0.477 (5)
H7BA	0.0908	0.2246	0.4426	0.051*	0.477 (5)
H7BB	0.0376	0.1702	0.4023	0.051*	0.477 (5)
H7BC	0.1022	0.0862	0.4072	0.051*	0.477 (5)
O1	0.08826 (13)	1.1645 (3)	0.15904 (12)	0.0444 (7)	
O2	0.09928 (10)	1.0439 (2)	0.07868 (9)	0.0276 (6)	
O3	0.23438 (9)	0.7396 (2)	0.19011 (8)	0.0198 (5)	
O5	0.27204 (11)	0.2696 (2)	0.37146 (9)	0.0297 (6)	
O6	0.30964 (17)	0.4278 (3)	0.43146 (12)	0.0604 (10)	
O7	0.32388 (10)	1.0796 (2)	0.06773 (9)	0.0204 (5)	
O8	0.37262 (11)	0.8752 (3)	0.04125 (10)	0.0323 (6)	
O9	0.07285 (10)	0.6385 (2)	0.22703 (9)	0.0234 (5)	
O10	0.05366 (12)	0.5113 (3)	0.14961 (10)	0.0360 (6)	
O11	0.05105 (10)	0.8403 (2)	0.14948 (9)	0.0257 (5)	
O1A	0.1440 (2)	0.2725 (5)	0.34135 (18)	0.0257 (11)	0.523 (5)
O2A	0.1551 (3)	0.3705 (6)	0.4252 (2)	0.0500 (17)	0.523 (5)
C14A	0.2939 (9)	0.4632 (14)	0.3083 (5)	0.022 (2)	0.523 (5)
H14D	0.3368	0.4602	0.3181	0.027*	0.523 (5)
O1B	0.1528 (3)	0.3714 (6)	0.3783 (2)	0.0365 (13)	0.477 (5)
O2B	0.1042 (2)	0.2270 (6)	0.3176 (2)	0.0369 (15)	0.477 (5)
C14B	0.2953 (10)	0.4398 (16)	0.2979 (6)	0.021 (2)	0.477 (5)
H14E	0.3383	0.4148	0.2966	0.026*	0.477 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.034 (2)	0.032 (2)	0.045 (2)	0.0022 (16)	0.0031 (17)	0.0078 (17)
C2	0.0195 (15)	0.0191 (15)	0.0214 (16)	0.0019 (12)	−0.0029 (13)	−0.0015 (12)
C3	0.0219 (15)	0.0173 (15)	0.0140 (14)	0.0006 (12)	0.0012 (12)	−0.0021 (12)
C4	0.0248 (16)	0.0150 (15)	0.0146 (14)	0.0011 (12)	0.0008 (12)	−0.0009 (12)
C5	0.0223 (16)	0.0197 (15)	0.0177 (15)	−0.0027 (13)	0.0044 (12)	0.0013 (12)
C9	0.046 (2)	0.0201 (18)	0.0264 (17)	−0.0003 (15)	−0.0106 (15)	0.0016 (14)
C10	0.037 (2)	0.062 (3)	0.076 (3)	0.017 (2)	0.003 (2)	0.018 (3)
C11	0.0264 (16)	0.0138 (15)	0.0181 (15)	−0.0002 (12)	−0.0015 (13)	0.0023 (12)
C12	0.0228 (16)	0.0127 (14)	0.0160 (14)	−0.0040 (12)	0.0019 (12)	−0.0034 (12)
C13	0.0247 (17)	0.0286 (19)	0.0145 (15)	−0.0010 (14)	0.0018 (13)	0.0011 (13)
C14	0.0236 (18)	0.044 (2)	0.0255 (18)	−0.0108 (16)	0.0051 (14)	−0.0027 (16)

C15	0.0185 (15)	0.0166 (15)	0.0184 (15)	0.0011 (12)	−0.0002 (12)	−0.0024 (12)
C16	0.0213 (16)	0.0193 (15)	0.0190 (15)	−0.0036 (12)	0.0044 (12)	0.0010 (12)
C17	0.0312 (18)	0.0279 (18)	0.0267 (18)	−0.0060 (15)	−0.0012 (15)	0.0064 (15)
C18	0.055 (3)	0.069 (3)	0.043 (2)	−0.033 (2)	0.005 (2)	0.007 (2)
C19	0.0195 (16)	0.0186 (15)	0.0178 (15)	−0.0004 (12)	−0.0004 (12)	−0.0040 (12)
C20	0.091 (3)	0.0222 (19)	0.0269 (19)	−0.004 (2)	−0.018 (2)	0.0061 (16)
C1A	0.033 (4)	0.015 (3)	0.016 (3)	−0.004 (3)	−0.005 (3)	0.000 (3)
C2A	0.026 (3)	0.018 (3)	0.020 (3)	−0.004 (2)	0.000 (2)	−0.001 (2)
C3A	0.023 (3)	0.018 (3)	0.022 (3)	−0.004 (3)	0.000 (3)	0.000 (2)
C4A	0.024 (4)	0.019 (4)	0.019 (4)	−0.003 (3)	0.000 (3)	−0.001 (3)
C5A	0.028 (3)	0.016 (4)	0.018 (4)	−0.005 (3)	−0.002 (4)	−0.003 (3)
C6A	0.034 (3)	0.029 (3)	0.027 (3)	−0.003 (3)	0.005 (3)	0.001 (3)
C7A	0.028 (6)	0.058 (7)	0.020 (5)	−0.014 (5)	0.014 (3)	0.002 (5)
C1B	0.031 (4)	0.015 (3)	0.017 (3)	−0.001 (3)	−0.006 (3)	−0.004 (3)
C2B	0.038 (3)	0.024 (3)	0.019 (3)	−0.011 (3)	−0.001 (3)	0.002 (3)
C3B	0.030 (3)	0.024 (3)	0.020 (3)	−0.008 (3)	0.001 (3)	−0.001 (3)
C4B	0.026 (4)	0.017 (4)	0.018 (4)	−0.007 (3)	0.001 (3)	0.001 (3)
C5B	0.029 (3)	0.015 (4)	0.018 (4)	−0.006 (3)	−0.001 (4)	−0.004 (3)
C6B	0.026 (3)	0.029 (3)	0.023 (3)	0.002 (3)	−0.001 (3)	0.005 (3)
C7B	0.025 (6)	0.050 (6)	0.026 (6)	−0.008 (5)	0.003 (4)	−0.006 (5)
O1	0.0471 (16)	0.0365 (16)	0.0497 (18)	0.0059 (13)	0.0068 (14)	−0.0009 (13)
O2	0.0228 (12)	0.0310 (13)	0.0290 (13)	0.0044 (10)	−0.0031 (10)	0.0084 (10)
O3	0.0189 (11)	0.0204 (11)	0.0200 (11)	0.0001 (9)	0.0016 (8)	0.0067 (9)
O5	0.0512 (15)	0.0180 (12)	0.0199 (11)	0.0003 (10)	−0.0083 (10)	0.0009 (9)
O6	0.112 (3)	0.0216 (15)	0.0476 (17)	−0.0083 (15)	−0.0493 (18)	0.0033 (13)
O7	0.0214 (11)	0.0161 (11)	0.0238 (11)	−0.0030 (9)	0.0033 (9)	0.0017 (9)
O8	0.0392 (14)	0.0251 (14)	0.0326 (14)	0.0042 (11)	0.0131 (11)	0.0013 (11)
O9	0.0208 (11)	0.0291 (12)	0.0205 (11)	−0.0060 (9)	0.0045 (9)	0.0025 (9)
O10	0.0475 (16)	0.0306 (14)	0.0299 (14)	−0.0111 (12)	0.0027 (12)	−0.0028 (11)
O11	0.0201 (12)	0.0267 (13)	0.0303 (13)	−0.0006 (9)	−0.0004 (10)	0.0032 (10)
O1A	0.030 (2)	0.024 (2)	0.024 (2)	−0.006 (2)	−0.002 (2)	0.0069 (18)
O2A	0.070 (4)	0.044 (3)	0.037 (3)	−0.029 (3)	0.023 (3)	−0.014 (3)
C14A	0.029 (3)	0.018 (4)	0.020 (4)	0.001 (3)	−0.007 (3)	−0.006 (3)
O1B	0.048 (3)	0.038 (3)	0.023 (3)	−0.018 (2)	0.000 (2)	0.009 (2)
O2B	0.029 (3)	0.037 (3)	0.045 (3)	−0.007 (2)	0.006 (3)	−0.005 (3)
C14B	0.029 (3)	0.017 (4)	0.018 (4)	0.002 (3)	−0.007 (3)	−0.005 (3)

Geometric parameters (Å, º)

C1—O1	1.200 (5)	C19—O11	1.233 (4)
C1—O2	1.383 (5)	C20—H20A	0.9600
C1—C10	1.497 (6)	C20—H20B	0.9600
C2—C11	1.371 (4)	C20—H20C	0.9600
C2—O2	1.391 (4)	C1A—O5	1.40 (2)
C2—C3	1.406 (4)	C1A—C2A	1.315 (18)
C3—C4	1.400 (4)	C1A—C14A	1.43 (3)
C3—C19	1.459 (4)	C2A—C3A	1.363 (9)
C4—O3	1.375 (4)	C2A—O1A	1.400 (7)

C4—C15	1.385 (4)	C3A—C4A	1.40 (3)
C5—C16	1.347 (4)	C3A—H3AA	0.9300
C5—O3	1.362 (4)	C4A—C5A	1.43 (4)
C5—C4B	1.49 (3)	C5A—C14A	1.38 (2)
C5—C4A	1.47 (3)	C5A—H5AA	0.9300
C9—O6	1.183 (4)	C6A—O2A	1.194 (7)
C9—O5	1.349 (4)	C6A—O1A	1.373 (7)
C9—C20	1.490 (5)	C6A—C7A	1.449 (10)
C10—H10A	0.9600	C7A—H7AA	0.9600
C10—H10B	0.9600	C7A—H7AB	0.9600
C10—H10C	0.9600	C7A—H7AC	0.9600
C11—C12	1.397 (4)	C1B—C14B	1.33 (3)
C11—H11	0.9300	C1B—C2B	1.44 (2)
C12—C15	1.368 (4)	C1B—O5	1.37 (3)
C12—O7	1.394 (4)	C2B—C3B	1.365 (11)
C13—O8	1.184 (4)	C2B—O1B	1.402 (7)
C13—O7	1.377 (4)	C3B—C4B	1.42 (3)
C13—C14	1.494 (4)	C3B—H3BA	0.9300
C14—H14A	0.9600	C4B—C5B	1.34 (5)
C14—H14B	0.9600	C5B—C14B	1.39 (2)
C14—H14C	0.9600	C5B—H5BA	0.9300
C15—H15	0.9300	C6B—O2B	1.197 (7)
C16—O9	1.395 (4)	C6B—O1B	1.371 (7)
C16—C19	1.456 (4)	C6B—C7B	1.450 (11)
C17—O10	1.191 (4)	C7B—H7BA	0.9600
C17—O9	1.386 (4)	C7B—H7BB	0.9600
C17—C18	1.486 (5)	C7B—H7BC	0.9600
C18—H18A	0.9600	C14A—H14D	0.9300
C18—H18B	0.9600	C14B—H14E	0.9300
C18—H18C	0.9600		

O1—C1—O2	122.4 (3)	H20B—C20—H20C	109.5
O1—C1—C10	127.0 (4)	O5—C1A—C2A	121.5 (15)
O2—C1—C10	110.6 (4)	O5—C1A—C14A	116.5 (14)
C11—C2—O2	117.6 (3)	C2A—C1A—C14A	122 (2)
C11—C2—C3	122.6 (3)	C3A—C2A—C1A	121.3 (11)
O2—C2—C3	119.9 (3)	C3A—C2A—O1A	117.1 (6)
C4—C3—C2	115.7 (3)	C1A—C2A—O1A	121.5 (11)
C4—C3—C19	119.7 (3)	C2A—C3A—C4A	121.3 (13)
C2—C3—C19	124.6 (3)	C2A—C3A—H3AA	119.4
O3—C4—C15	114.7 (3)	C4A—C3A—H3AA	119.4
O3—C4—C3	121.5 (3)	C5A—C4A—C3A	117 (2)
C15—C4—C3	123.8 (3)	C5A—C4A—C5	118 (2)
C16—C5—O3	119.9 (3)	C3A—C4A—C5	124 (2)
C16—C5—C4B	131.4 (13)	C14A—C5A—C4A	121 (2)
O3—C5—C4B	108.7 (12)	C14A—C5A—H5AA	119.7
C16—C5—C4A	127.8 (12)	C4A—C5A—H5AA	119.7
O3—C5—C4A	112.3 (12)	O2A—C6A—O1A	121.8 (6)

O6—C9—O5	122.2 (3)	O2A—C6A—C7A	118.6 (9)
O6—C9—C20	127.3 (3)	O1A—C6A—C7A	118.4 (9)
O5—C9—C20	110.5 (3)	C6A—C7A—H7AA	109.5
C1—C10—H10A	109.5	C6A—C7A—H7AB	109.5
C1—C10—H10B	109.5	H7AA—C7A—H7AB	109.5
H10A—C10—H10B	109.5	C6A—C7A—H7AC	109.5
C1—C10—H10C	109.5	H7AA—C7A—H7AC	109.5
H10A—C10—H10C	109.5	H7AB—C7A—H7AC	109.5
H10B—C10—H10C	109.5	C14B—C1B—C2B	116 (2)
C2—C11—C12	118.1 (3)	C14B—C1B—O5	129.2 (16)
C2—C11—H11	120.9	C2B—C1B—O5	114.0 (16)
C12—C11—H11	120.9	C3B—C2B—C1B	122.1 (12)
C15—C12—O7	121.5 (3)	C3B—C2B—O1B	119.7 (7)
C15—C12—C11	122.7 (3)	C1B—C2B—O1B	118.1 (12)
O7—C12—C11	115.6 (3)	C2B—C3B—C4B	118.3 (15)
O8—C13—O7	123.2 (3)	C2B—C3B—H3BA	120.8
O8—C13—C14	126.5 (3)	C4B—C3B—H3BA	120.8
O7—C13—C14	110.3 (3)	C5B—C4B—C3B	119 (3)
C13—C14—H14A	109.5	C5B—C4B—C5	122 (3)
C13—C14—H14B	109.5	C3B—C4B—C5	119 (2)
H14A—C14—H14B	109.5	C4B—C5B—C14B	122 (2)
C13—C14—H14C	109.5	C4B—C5B—H5BA	119.2
H14A—C14—H14C	109.5	C14B—C5B—H5BA	119.2
H14B—C14—H14C	109.5	O2B—C6B—O1B	121.4 (7)
C12—C15—C4	117.1 (3)	O2B—C6B—C7B	118.7 (9)
C12—C15—H15	121.4	O1B—C6B—C7B	118.0 (10)
C4—C15—H15	121.4	C6B—C7B—H7BA	109.5
C5—C16—O9	120.2 (3)	C6B—C7B—H7BB	109.5
C5—C16—C19	124.1 (3)	H7BA—C7B—H7BB	109.5
O9—C16—C19	115.5 (3)	C6B—C7B—H7BC	109.5
O10—C17—O9	122.4 (3)	H7BA—C7B—H7BC	109.5
O10—C17—C18	127.8 (3)	H7BB—C7B—H7BC	109.5
O9—C17—C18	109.7 (3)	C1—O2—C2	115.5 (3)
C17—C18—H18A	109.5	C5—O3—C4	121.0 (2)
C17—C18—H18B	109.5	C9—O5—C1A	122.1 (6)
H18A—C18—H18B	109.5	C9—O5—C1B	116.0 (6)
C17—C18—H18C	109.5	C13—O7—C12	119.1 (2)
H18A—C18—H18C	109.5	C17—O9—C16	115.3 (2)
H18B—C18—H18C	109.5	C6A—O1A—C2A	117.3 (5)
O11—C19—C16	121.0 (3)	C5A—C14A—C1A	117.7 (17)
O11—C19—C3	125.2 (3)	C5A—C14A—H14D	121.1
C16—C19—C3	113.7 (3)	C1A—C14A—H14D	121.1
C9—C20—H20A	109.5	C6B—O1B—C2B	116.8 (6)
C9—C20—H20B	109.5	C1B—C14B—C5B	122.4 (19)
H20A—C20—H20B	109.5	C1B—C14B—H14E	118.8
C9—C20—H20C	109.5	C5B—C14B—H14E	118.8
H20A—C20—H20C	109.5		

C6A—O1A—C2A—C1A	67.6 (12)	O2—C2—C11—C12	−179.0 (3)
C6A—O1A—C2A—C3A	−115.3 (7)	O2—C2—C3—C19	0.5 (4)
C2A—O1A—C6A—C7A	163.8 (8)	O2—C2—C3—C4	179.7 (3)
C2A—O1A—C6A—O2A	−3.5 (9)	C1A—C2A—C3A—C4A	1 (2)
C6B—O1B—C2B—C1B	−106.1 (11)	O1A—C2A—C3A—C4A	−176.5 (15)
C6B—O1B—C2B—C3B	77.2 (9)	C2—C3—C19—O11	0.2 (5)
C2B—O1B—C6B—C7B	161.1 (10)	C4—C3—C19—C16	1.6 (4)
C2B—O1B—C6B—O2B	−3.1 (11)	C2—C3—C19—C16	−179.3 (3)
C2—O2—C1—C10	−176.3 (3)	C19—C3—C4—C15	177.5 (3)
C1—O2—C2—C11	−108.2 (3)	C2—C3—C4—O3	178.2 (3)
C2—O2—C1—O1	4.3 (5)	C19—C3—C4—O3	−2.6 (4)
C1—O2—C2—C3	74.1 (3)	C2—C3—C4—C15	−1.7 (4)
C5—O3—C4—C15	−179.0 (2)	C4—C3—C19—O11	−179.0 (3)
C4—O3—C5—C4A	178.5 (12)	C2A—C3A—C4A—C5A	2 (3)
C4—O3—C5—C16	1.5 (4)	C2A—C3A—C4A—C5	−175.7 (15)
C5—O3—C4—C3	1.1 (4)	O3—C4—C15—C12	−179.2 (2)
C9—O5—C1A—C14A	70.9 (16)	C3—C4—C15—C12	0.7 (4)
C1A—O5—C9—C20	163.8 (8)	C5A—C4A—C5—C16	−173.0 (13)
C1A—O5—C9—O6	−16.3 (9)	C3A—C4A—C5A—C14A	−3 (3)
C9—O5—C1A—C2A	−115.6 (14)	C5A—C4A—C5—O3	10 (2)
C13—O7—C12—C11	−131.8 (3)	C3A—C4A—C5—C16	5 (3)
C12—O7—C13—C14	−176.9 (3)	C3A—C4A—C5—O3	−171.8 (18)
C12—O7—C13—O8	4.6 (4)	C5—C4A—C5A—C14A	175.2 (16)
C13—O7—C12—C15	52.9 (4)	C4A—C5—C16—C19	−179.0 (14)
C17—O9—C16—C5	−110.5 (3)	C4A—C5—C16—O9	5.6 (15)
C17—O9—C16—C19	73.7 (3)	O3—C5—C16—C19	−2.6 (4)
C16—O9—C17—C18	−178.6 (3)	O3—C5—C16—O9	−178.0 (2)
C16—O9—C17—O10	3.9 (5)	C4A—C5A—C14A—C1A	1 (3)
O5—C1A—C14A—C5A	175.5 (13)	C2—C11—C12—C15	0.2 (4)
C14A—C1A—C2A—C3A	−3 (2)	C2—C11—C12—O7	−175.1 (3)
O5—C1A—C2A—O1A	1 (2)	O7—C12—C15—C4	175.1 (3)
C2A—C1A—C14A—C5A	2 (2)	C11—C12—C15—C4	0.1 (4)
O5—C1A—C2A—C3A	−176.0 (10)	O9—C16—C19—O11	−2.9 (4)
C14A—C1A—C2A—O1A	174.2 (12)	C5—C16—C19—C3	1.0 (4)
C11—C2—C3—C19	−177.2 (3)	O9—C16—C19—C3	176.6 (2)
C11—C2—C3—C4	2.0 (4)	C5—C16—C19—O11	−178.5 (3)
C3—C2—C11—C12	−1.3 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3*A*—H3*AA*···O9	0.93	2.22	2.842 (7)	124
C5*A*—H5*AA*···O3	0.93	2.32	2.675 (18)	102
C7*A*—H7*AB*···O8^{i}	0.96	2.43	3.331 (17)	156
C20—H20*A*···O2*A*^{ii}	0.96	2.33	3.142 (7)	142
C20—H20*B*···O6^{ii}	0.96	2.51	3.352 (5)	146

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