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Synthesis, crystal structure and Hirshfeld surface analysis of naphthalene-2,3-diyl bis(3-benzyloxy)-benzoate

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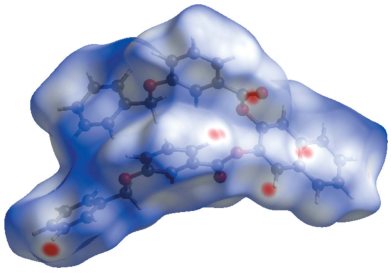
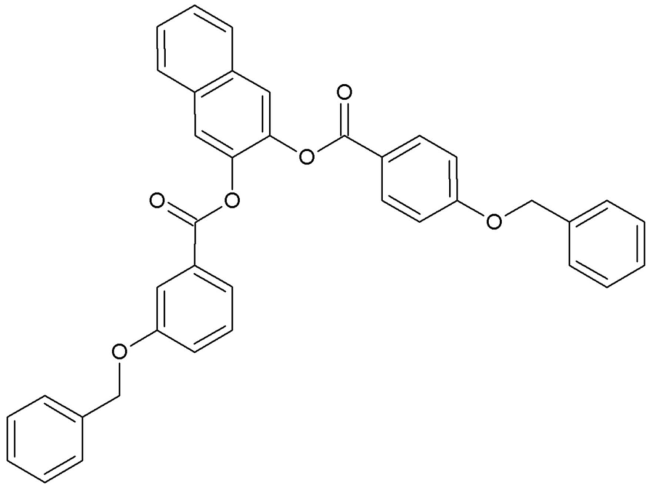
In the title compound, $C_{38}H_{28}O_6$, the dihedral angles between the naphthalene ring system and its pendant benzyloxy rings *A* and *B* are 88.05 (7) and 80.84 (7)°, respectively. The dihedral angles between the *A* and *B* rings and their attached phenyl rings are 49.15 (8) and 80.78 (8)°, respectively. In the extended structure, the molecules are linked by weak C—H···O and C—H···π hydrogen bonds, and π–π stacking interactions, which variously generate $C(11)$ chains and $R_2^2(12)$ loops as part of a three-dimensional network. The Hirshfeld surface [fingerprint contributions = H···H (42.3%), C···H/H···C (40.3%) and O···H/H···O (15.7%)] and intermolecular interaction energies are reported, with dispersion (E_{dis} = −428.6 kJ mol^{-1}) being the major contributor.

1. Chemical context

Naphthalene, biphenyl or benzene rings can act as rigid cores in liquid crystal molecules. A variety of banana-shaped, bow-shaped or bent-core ferroelectric liquid crystals were developed by incorporating a benzene ring as a rigid core (Noiri *et al.*, 1996; Srinivasa *et al.*, 2017). These types of compounds form lamellar and/or columnar mesophases (Szydlowska *et al.*, 2003) and they have been subjected to experimental and theoretical studies (Reddy *et al.*, 2006; Vaupotič, 2006). Liquid crystalline materials with a bent-core molecule are attractive because they exhibit good physical properties and possess two-dimensional smectic phases that display qualitatively different physical properties than calamatic ferroelectric liquid crystals.





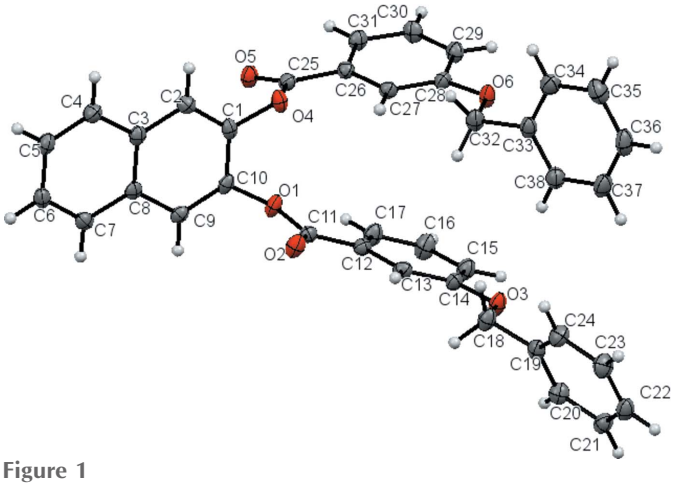


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

Our team is studying new bent-core liquid crystals with naphthalene rings as a rigid core (Srinivasa *et al.*, 2018) and, as part of that work, we have performed a simple coupling reaction between 1,2-dihydroxynaphthalene and 3-benzyloxybenzoic acid to construct the title molecule. It is a bent-type non-liquid crystal material, possibly due to the absence of alkyl chains/polar moiety at the ends of the molecule.

2. Structural commentary

The title compound crystallizes with one molecule in the asymmetric unit (Fig. 1) in the space group $P2_1/n$. The dihedral angles between the C1–C10 naphthalene ring system (r.m.s. deviation = 0.022 Å) and its pendant C26–C31 (*A*) and C12–C17 (*B*) benzyloxy rings are 88.05 (7) and 80.84 (7)°, respectively. The dihedral angles between the *A* and *B* rings and their attached C33–C38 and C19–C24 phenyl rings are 49.15 (8) and 80.78 (8)°, respectively. Key torsion angles include C1—O4—C25—C26 [−160.98 (13)°], C28—O2—C32—C33 [−172.04 (14)°], C10—O1—C11—C12 [−168.94 (14)°] and C14—O3—C18—C19 [172.84 (14)°]. Otherwise, the geometrical data for the title compound may be regarded as normal.

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

*Cg*1, *Cg*2, *Cg*4, *Cg*5, *Cg*6 and *Cg*7 are the centroids of the C1–C3/C8–C10, C3–C8, C19–C24, C26–C31, C33–C38 and C1–C10 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···O5^{i}	0.93	2.52	3.258 (2)	136
C21—H21···O2^{ii}	0.93	2.49	3.378 (2)	160
C4—H4···*Cg*5^{iii}	0.93	2.60	3.4949 (19)	163
C16—H16···*Cg*2^{i}	0.93	2.95	3.6955 (18)	139
C17—H17···*Cg*1^{i}	0.93	2.90	3.7480 (17)	152
C17—H17···*Cg*7^{i}	0.93	2.91	3.6342 (17)	135
C18—H18*A*···*Cg*6^{ii}	0.97	2.66	3.5201 (18)	148
C30—H30···*Cg*1^{iv}	0.93	2.90	3.7078 (17)	146
C31—H31···*Cg*2^{iv}	0.93	2.69	3.5449 (17)	154
C31—H31···*Cg*7^{iv}	0.93	2.95	3.6130 (16)	130
C32—H32*A*···*Cg*4^{v}	0.97	2.82	3.5525 (18)	133
C15—H15···*Cg*6^{vi}	0.93	2.97	3.6860 (18)	135

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+1, -y, -z$; (iv) $x-1, y, z$; (v) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (vi) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

3. Supramolecular features

In the crystal, the molecules are linked by numerous C—H···O and C—H···π interactions (Table 1). Prominent packing features include a *C*(11) chain (arising from the C21—H21···O2^{ii} hydrogen bond), which runs along [010], and centrosymmetric $R_2^2(12)$ loops (arising from the C9—H9···O5^{i} hydrogen bond) between the molecules as shown in Fig. 2. These, and the C—H···π interactions, link the molecules into a three-dimensional network (see Figs. S1 and S2 in the supporting information).

4. Hirshfeld surface analysis

The title molecule was subjected to Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) and the two-dimensional (2D) fingerprint plots (McKinnon *et al.*, 2007) were generated with *CrystalExplorer17* (Turner *et al.*, 2017). The Hirshfeld surface mapped on $d_{\rm norm}$ is shown in Fig. 3 and the overall 2D fingerprint plot and those delineated into H···H (42.3%), C···H/H···C (40.3%) and O···H/H···O (15.7%) contacts, together with their relative contributions to the Hirshfeld surface, are illustrated in Fig. 4. The interaction energies for

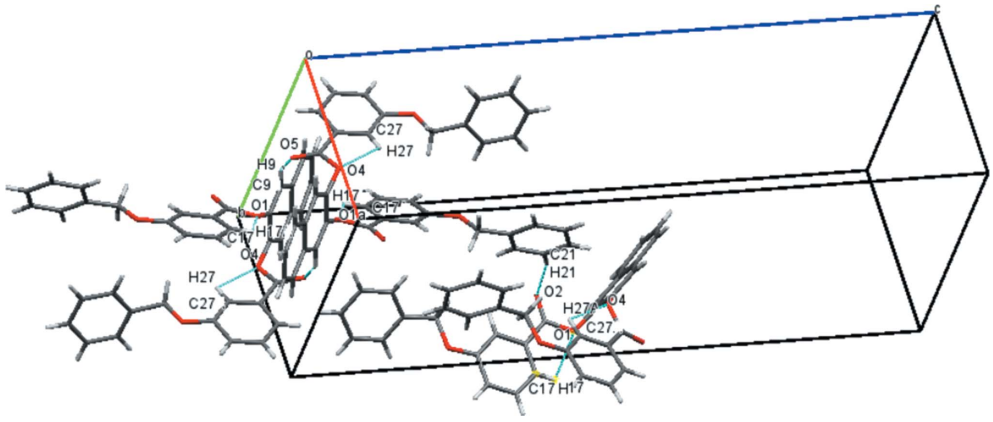


Figure 2
Partial packing diagram showing the C—H···O interactions.

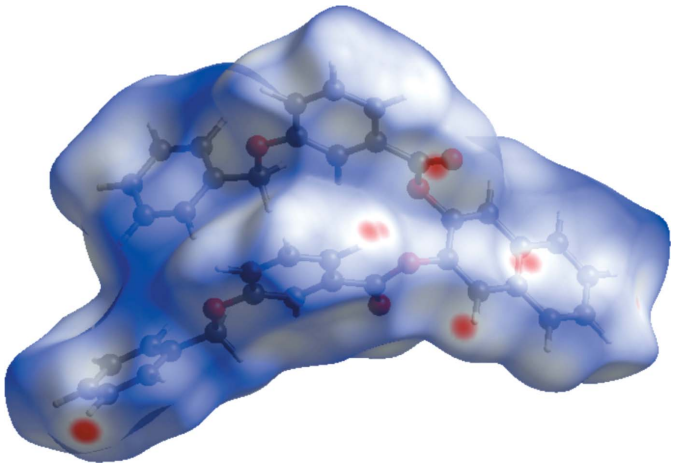


Figure 3
The Hirshfeld surface of the title compound mapped over d_{norm}.

the title compound were calculated at the HF/3-21G quantum level of theory in *CrystalExplorer*. The four energy variables that make up the total intermolecular interaction energy (E_{tot}) are electrostatic (E_{ele}), polarization (E_{pol}), dispersion (E_{disp}) and exchange–repulsion (E_{rep}), and the cylinder-shaped energy frameworks represent the relative strengths of the interaction energies in individual directions, as well as the topologies of pairwise intermolecular interaction energies within the crystal (Mackenzie *et al.*, 2017). The energies between molecular pairs are depicted as cylinders connecting the centroids of two molecules, with the radius of the cylinder equal to the amount of interaction energy between the molecules (Wu *et al.*, 2020). The net interaction energies for the title compound are E_{ele} = −56.3 kJ mol^{-1}, E_{pol} = −30.4.0 kJ mol^{-1}, E_{dis} = −428.6 kJ mol^{-1} and E_{rep} = 160.4 kJ mol^{-1}, with a total interaction energy E_{tot} of −333.3 kJ mol^{-1}. Therefore, E_{dis} is the major interaction energy in the title compound. The energy framework showing the electrostatic potential force, dispersion force and total energy diagrams are shown in Fig. 5. The cylindrical radii are proportional to the relative strength of the corresponding energies and they were adjusted to the same scale factor of 50 with a cutoff value of 5 kJ mol^{-1}.

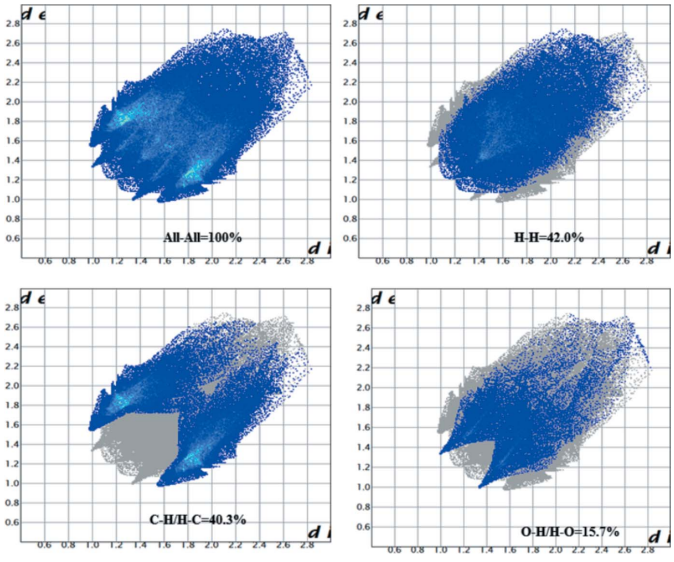


Figure 4
The 2D fingerprint plots for the title compound, showing C···H/H···C, H···H/H···H, O···H/H···O and O···O/O···O contacts.

5. Database survey

A search of the Cambridge Structural Database (CSD; Version 5.43, update of March 2022: Groom *et al.*, 2016) for the naphthalene-2,3-diyl fragment gave 26 hits, of which six molecules are similar to the title compound, with CSD refcodes WAFRII, WAFROO, WAFRUU, WAFSAB,

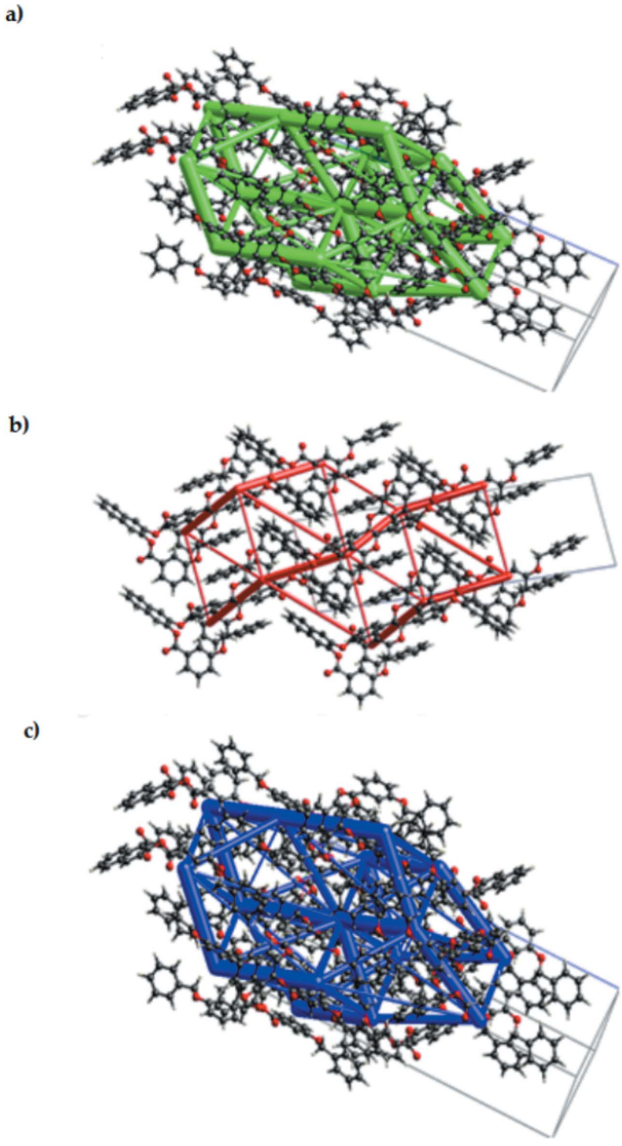


Figure 5
Energy frameworks calculated for the title compound, viewed along the *a*-axis direction, showing (*a*) Coulomb potential force, (*b*) dispersion force and (*c*) total energy diagrams. The cylindrical radii are proportional to the relative strength of the corresponding energies and they were adjusted to a cutoff value of 5 kJ mol^{-1}.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{38}H_{28}O_6$
M_r	580.60
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	302
a, b, c (Å)	9.5219 (2), 10.1010 (2), 30.7050 (8)
β (°)	96.666 (1)
V (Å^3)	2933.26 (11)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.72
Crystal size (mm)	0.32 × 0.28 × 0.21
Data collection	
Diffractometer	Bruker SMART APEXII CCD
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14741, 4775, 4298
R_{int}	0.032
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.585
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.142, 1.06
No. of reflections	4775
No. of parameters	397
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.23

Computer programs: *APEX3* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *Mercury* (Macrae *et al.*, 2020).

WAFSEF and WAFSIJ (Rutherford *et al.*, 2020). There exist intermolecular interactions dominated by π–π stacking and C—H···π interactions involving the arene rings in the benzoate fragments and the arene ring in the tetrahydronapthalene moiety. A 'thermosalient phase transition effect' was studied in the compounds coded QIBMUM and QIBMUM01–QIBMUM06 (Tamboli *et al.*, 2013), which feature a naphthalene-2,3-diyl bis(4-fluorobenzoate) fragment. The presence of π–π stacking and C—H···O and C—H···F interactions appear to play an important role in determining the molecular conformations. The crystal structure analyses of the polymorphic structures coded DOPPAB, DOPPAB01, DOPPAB02, DOPPOP, DOPPOP01 and DOPQAC (Tamboli *et al.*, 2018) revealed weak intermolecular interactions, such as C—H···O, C—H···π and π–π stacking, as also seen in the title molecule. These interactions are actively involved in molecular aggregation, which results in the polymorphic modifications, if they are subjected to thermal transformation. Here, all the molecules crystallize in the space group *Pbcn* or *P*2/*c*. The crystal structure analyses of IJAGIJ01 to IJAGIJ05 (Tamboli *et al.*, 2014) are polymorphs of isomeric napthalene-2,3-diol ditoluates, in which the intermolecular interactions, such as C—H···O, C—H···π and π–π stacking, are similar to the interactions present in the title molecule.

6. Synthesis and crystalization

Under an inert atmosphere, 1,2-dihydroxynaphthalene (1.00 mmol), a catalytic amount of 4-dimethylaminopyridine and 3-benzyloxybenzoic acid (2.00 mmol) were dissolved in 50 ml of dry dichloromethane (DCM). The above mixture was stirred for 2 h at room temperature with a solution of *N*,*N*-dicyclohexylcarbodiimide (1.2 mmol) in DCM (20 ml). Filtration was used to remove the precipitated *N*,*N*-dicyclohexylurea and the solvent was evaporated. To obtain the pure product, the solid residue was purified using column chromatography on silica gel with DCM as an eluent, followed by recrystallization from ethyl alcohol solution.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were positioned geometrically (C—H = 0.93 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

Acknowledgements

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supporting information

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Computing details

Data collection: *APEX3* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *SHELXL* (Sheldrick, 2015b).

Naphthalene-2,3-diyl bis(3-benzyloxy)benzoate

Crystal data

$C_{38}H_{28}O_6$
M_r = 580.60
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 9.5219 (2) Å
b = 10.1010 (2) Å
c = 30.7050 (8) Å
β = 96.666 (1)°
V = 2933.26 (11) Å^3
Z = 4
$F(000)$ = 1216
Prism
D_x = 1.315 Mg m^{-3}
Melting point: 417 K
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 4775 reflections
θ = 0.3–25°
μ = 0.72 mm^{-1}
T = 302 K
Rod, colourless
0.32 × 0.28 × 0.21 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 2.06 pixels mm^{-1}
ω scans
14741 measured reflections
4775 independent reflections
4298 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 64.5°, θ_{min} = 4.6°
h = −11→10
k = −11→7
l = −32→35

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.142
S = 1.06
4775 reflections
397 parameters
0 restraints
1 constraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0997P)^2 + 0.5664P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.58939 (11)	0.43382 (12)	0.05018 (3)	0.0232 (3)
O2	0.77634 (11)	0.38287 (13)	0.09926 (3)	0.0272 (3)
O3	0.58453 (11)	0.47609 (13)	0.24604 (3)	0.0268 (3)
O4	0.50928 (11)	0.18476 (12)	0.02981 (3)	0.0225 (3)
O5	0.34497 (11)	0.27300 (12)	−0.02087 (3)	0.0248 (3)
O6	0.25879 (11)	0.13423 (13)	0.16475 (3)	0.0253 (3)
C1	0.61513 (15)	0.23968 (17)	0.00711 (5)	0.0201 (4)
C2	0.67835 (16)	0.16830 (17)	−0.02259 (5)	0.0213 (4)
H2	0.649084	0.082186	−0.029440	0.026*
C3	0.78971 (15)	0.22628 (17)	−0.04315 (5)	0.0204 (4)
C4	0.86373 (17)	0.15418 (18)	−0.07275 (5)	0.0243 (4)
H4	0.838208	0.067021	−0.079451	0.029*
C5	0.97232 (17)	0.21079 (18)	−0.09165 (5)	0.0254 (4)
H5	1.021228	0.161481	−0.110563	0.031*
C6	1.01010 (16)	0.34325 (18)	−0.08256 (5)	0.0237 (4)
H6	1.083712	0.381211	−0.095640	0.028*
C7	0.93960 (15)	0.41691 (18)	−0.05465 (5)	0.0212 (4)
H7	0.963984	0.505156	−0.049498	0.025*
C8	0.82939 (15)	0.35947 (17)	−0.03349 (4)	0.0187 (3)
C9	0.76033 (15)	0.43085 (17)	−0.00204 (5)	0.0194 (3)
H9	0.785118	0.518167	0.004687	0.023*
C10	0.65814 (15)	0.37016 (17)	0.01796 (4)	0.0200 (4)
C11	0.65782 (15)	0.42547 (17)	0.09173 (5)	0.0199 (4)
C12	0.56915 (16)	0.47090 (16)	0.12536 (5)	0.0203 (4)
C13	0.62360 (16)	0.45161 (17)	0.16931 (5)	0.0213 (4)
H13	0.712355	0.413805	0.176414	0.026*
C14	0.54323 (16)	0.48970 (17)	0.20201 (5)	0.0216 (4)
C15	0.40898 (17)	0.54473 (18)	0.19114 (5)	0.0266 (4)
H15	0.354379	0.568536	0.213113	0.032*
C16	0.35781 (17)	0.5636 (2)	0.14761 (5)	0.0307 (4)
H16	0.269350	0.601978	0.140482	0.037*
C17	0.43662 (17)	0.52605 (19)	0.11437 (5)	0.0272 (4)
H17	0.400928	0.537764	0.085129	0.033*
C18	0.72408 (17)	0.42395 (19)	0.25874 (5)	0.0265 (4)
H18A	0.793380	0.474299	0.244862	0.032*
H18B	0.728805	0.332338	0.249542	0.032*
C19	0.75487 (16)	0.43351 (18)	0.30777 (5)	0.0234 (4)

C20	0.73388 (16)	0.55202 (18)	0.32920 (5)	0.0250 (4)
H20	0.696094	0.624617	0.313295	0.030*
C21	0.76914 (16)	0.56235 (19)	0.37426 (5)	0.0262 (4)
H21	0.754780	0.641792	0.388404	0.031*
C22	0.82545 (17)	0.45503 (19)	0.39814 (5)	0.0282 (4)
H22	0.849553	0.462230	0.428278	0.034*
C23	0.84571 (19)	0.3374 (2)	0.37712 (5)	0.0316 (4)
H23	0.883045	0.264814	0.393102	0.038*
C24	0.81042 (18)	0.32703 (19)	0.33198 (5)	0.0286 (4)
H24	0.824505	0.247257	0.318000	0.034*
C25	0.37343 (15)	0.22511 (16)	0.01482 (5)	0.0192 (3)
C26	0.27380 (16)	0.20240 (16)	0.04756 (5)	0.0194 (3)
C27	0.32213 (16)	0.17695 (16)	0.09144 (5)	0.0199 (3)
H27	0.418421	0.168975	0.100463	0.024*
C28	0.22447 (16)	0.16371 (17)	0.12146 (5)	0.0204 (4)
C29	0.08046 (16)	0.17887 (18)	0.10773 (5)	0.0235 (4)
H29	0.015499	0.173477	0.128056	0.028*
C30	0.03403 (16)	0.20179 (18)	0.06419 (5)	0.0250 (4)
H30	−0.062326	0.209607	0.055252	0.030*
C31	0.12965 (16)	0.21333 (17)	0.03353 (5)	0.0233 (4)
H31	0.098083	0.228097	0.004124	0.028*
C32	0.40533 (16)	0.1048 (2)	0.17832 (5)	0.0267 (4)
H32A	0.462305	0.183689	0.176271	0.032*
H32B	0.438343	0.037409	0.159440	0.032*
C33	0.41890 (16)	0.05652 (19)	0.22477 (5)	0.0247 (4)
C34	0.39745 (18)	−0.0760 (2)	0.23388 (5)	0.0311 (4)
H34	0.369988	−0.134322	0.211041	0.037*
C35	0.41657 (19)	−0.1225 (2)	0.27679 (6)	0.0350 (4)
H35	0.401233	−0.211366	0.282630	0.042*
C36	0.45865 (18)	−0.0357 (2)	0.31084 (5)	0.0331 (4)
H36	0.473278	−0.066367	0.339563	0.040*
C37	0.47870 (18)	0.0964 (2)	0.30192 (5)	0.0330 (4)
H37	0.506345	0.154630	0.324773	0.040*
C38	0.45797 (17)	0.1431 (2)	0.25914 (5)	0.0281 (4)
H38	0.470290	0.232544	0.253504	0.034*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0207 (5)	0.0346 (7)	0.0141 (5)	0.0060 (5)	0.0012 (4)	−0.0030 (4)
O2	0.0200 (6)	0.0404 (8)	0.0207 (6)	0.0065 (5)	−0.0001 (4)	−0.0027 (5)
O3	0.0203 (6)	0.0440 (8)	0.0159 (5)	0.0045 (5)	0.0003 (4)	−0.0018 (5)
O4	0.0168 (5)	0.0302 (7)	0.0212 (5)	0.0025 (4)	0.0048 (4)	0.0067 (5)
O5	0.0230 (6)	0.0345 (7)	0.0167 (6)	0.0007 (5)	0.0013 (4)	0.0025 (5)
O6	0.0190 (6)	0.0417 (8)	0.0151 (5)	0.0018 (5)	0.0023 (4)	0.0017 (5)
C1	0.0150 (7)	0.0286 (10)	0.0167 (7)	0.0005 (6)	0.0015 (5)	0.0053 (6)
C2	0.0201 (8)	0.0218 (9)	0.0217 (8)	−0.0004 (6)	0.0013 (6)	0.0019 (6)
C3	0.0181 (7)	0.0276 (10)	0.0151 (7)	0.0010 (6)	−0.0002 (6)	0.0012 (6)

C4	0.0255 (8)	0.0243 (10)	0.0233 (8)	−0.0003 (7)	0.0036 (6)	−0.0029 (7)
C5	0.0225 (8)	0.0342 (11)	0.0203 (8)	0.0023 (7)	0.0050 (6)	−0.0025 (7)
C6	0.0182 (7)	0.0352 (11)	0.0176 (7)	−0.0031 (7)	0.0021 (6)	0.0023 (7)
C7	0.0198 (8)	0.0247 (9)	0.0179 (7)	−0.0027 (6)	−0.0025 (6)	0.0027 (6)
C8	0.0165 (7)	0.0250 (9)	0.0135 (7)	−0.0003 (6)	−0.0025 (5)	0.0008 (6)
C9	0.0184 (7)	0.0223 (9)	0.0163 (7)	0.0014 (6)	−0.0023 (6)	−0.0015 (6)
C10	0.0172 (7)	0.0293 (10)	0.0127 (7)	0.0045 (6)	−0.0011 (5)	−0.0018 (6)
C11	0.0191 (8)	0.0226 (9)	0.0174 (7)	−0.0012 (6)	−0.0003 (6)	−0.0003 (6)
C12	0.0196 (8)	0.0224 (9)	0.0187 (7)	−0.0014 (6)	0.0014 (6)	−0.0019 (6)
C13	0.0170 (7)	0.0244 (9)	0.0222 (8)	−0.0001 (6)	0.0005 (6)	−0.0021 (6)
C14	0.0221 (8)	0.0272 (9)	0.0154 (7)	−0.0031 (7)	0.0010 (6)	−0.0021 (6)
C15	0.0213 (8)	0.0364 (11)	0.0226 (8)	0.0026 (7)	0.0041 (6)	−0.0051 (7)
C16	0.0202 (8)	0.0458 (12)	0.0256 (8)	0.0096 (7)	0.0008 (6)	−0.0015 (7)
C17	0.0243 (8)	0.0377 (11)	0.0185 (7)	0.0042 (7)	−0.0015 (6)	−0.0001 (7)
C18	0.0207 (8)	0.0391 (11)	0.0197 (8)	0.0048 (7)	0.0015 (6)	−0.0025 (7)
C19	0.0159 (7)	0.0341 (10)	0.0200 (8)	−0.0002 (7)	0.0018 (6)	−0.0012 (7)
C20	0.0219 (8)	0.0292 (10)	0.0236 (8)	0.0039 (7)	0.0013 (6)	0.0014 (7)
C21	0.0205 (8)	0.0339 (11)	0.0244 (8)	0.0000 (7)	0.0035 (6)	−0.0071 (7)
C22	0.0235 (8)	0.0430 (12)	0.0180 (8)	−0.0026 (7)	0.0015 (6)	−0.0003 (7)
C23	0.0358 (10)	0.0349 (11)	0.0235 (8)	0.0033 (8)	0.0011 (7)	0.0057 (7)
C24	0.0312 (9)	0.0290 (10)	0.0258 (8)	0.0025 (7)	0.0046 (7)	−0.0029 (7)
C25	0.0188 (7)	0.0207 (9)	0.0178 (7)	−0.0004 (6)	0.0013 (6)	−0.0023 (6)
C26	0.0193 (8)	0.0200 (9)	0.0190 (8)	0.0004 (6)	0.0027 (6)	−0.0011 (6)
C27	0.0160 (7)	0.0235 (9)	0.0199 (8)	−0.0003 (6)	0.0010 (6)	−0.0016 (6)
C28	0.0222 (8)	0.0231 (9)	0.0157 (7)	−0.0009 (6)	0.0017 (6)	−0.0018 (6)
C29	0.0187 (8)	0.0291 (10)	0.0234 (8)	0.0019 (6)	0.0054 (6)	−0.0003 (7)
C30	0.0156 (7)	0.0333 (10)	0.0258 (8)	0.0033 (7)	0.0017 (6)	0.0023 (7)
C31	0.0217 (8)	0.0277 (10)	0.0198 (7)	0.0013 (7)	−0.0004 (6)	0.0019 (6)
C32	0.0186 (8)	0.0416 (11)	0.0195 (8)	−0.0022 (7)	0.0006 (6)	0.0021 (7)
C33	0.0176 (7)	0.0379 (11)	0.0188 (8)	−0.0019 (7)	0.0025 (6)	0.0009 (7)
C34	0.0296 (9)	0.0386 (12)	0.0250 (9)	−0.0057 (8)	0.0032 (7)	−0.0028 (7)
C35	0.0330 (9)	0.0406 (12)	0.0319 (9)	−0.0028 (8)	0.0064 (7)	0.0098 (8)
C36	0.0279 (9)	0.0519 (13)	0.0195 (8)	0.0004 (8)	0.0030 (6)	0.0083 (8)
C37	0.0308 (9)	0.0491 (13)	0.0189 (8)	−0.0036 (8)	0.0021 (7)	−0.0025 (8)
C38	0.0247 (8)	0.0362 (11)	0.0235 (8)	−0.0024 (7)	0.0034 (6)	0.0006 (7)

Geometric parameters (Å, º)

O1—C11	1.3662 (18)	C18—C19	1.503 (2)
O1—C10	1.4040 (18)	C18—H18A	0.9700
O2—C11	1.2049 (19)	C18—H18B	0.9700
O3—C14	1.3702 (18)	C19—C24	1.378 (3)
O3—C18	1.4404 (19)	C19—C20	1.392 (2)
O4—C25	1.3831 (18)	C20—C21	1.389 (2)
O4—C1	1.4045 (18)	C20—H20	0.9300
O5—C25	1.1998 (18)	C21—C22	1.382 (3)
O6—C28	1.3642 (18)	C21—H21	0.9300
O6—C32	1.4401 (18)	C22—C23	1.376 (3)

C1—C2	1.357 (2)	C22—H22	0.9300
C1—C10	1.409 (2)	C23—C24	1.392 (2)
C2—C3	1.422 (2)	C23—H23	0.9300
C2—H2	0.9300	C24—H24	0.9300
C3—C4	1.415 (2)	C25—C26	1.478 (2)
C3—C8	1.419 (2)	C26—C31	1.394 (2)
C4—C5	1.368 (2)	C26—C27	1.396 (2)
C4—H4	0.9300	C27—C28	1.390 (2)
C5—C6	1.405 (3)	C27—H27	0.9300
C5—H5	0.9300	C28—C29	1.395 (2)
C6—C7	1.368 (2)	C29—C30	1.378 (2)
C6—H6	0.9300	C29—H29	0.9300
C7—C8	1.421 (2)	C30—C31	1.388 (2)
C7—H7	0.9300	C30—H30	0.9300
C8—C9	1.425 (2)	C31—H31	0.9300
C9—C10	1.356 (2)	C32—C33	1.498 (2)
C9—H9	0.9300	C33—C34	1.387 (3)
C11—C12	1.480 (2)	C33—C38	1.388 (2)
C12—C17	1.385 (2)	C34—C35	1.391 (2)
C12—C13	1.402 (2)	C34—H34	0.9300
C13—C14	1.386 (2)	C35—C36	1.388 (3)
C13—H13	0.9300	C35—H35	0.9300
C14—C15	1.398 (2)	C36—C37	1.379 (3)
C15—C16	1.382 (2)	C36—H36	0.9300
C15—H15	0.9300	C37—C38	1.388 (2)
C16—C17	1.388 (2)	C37—H37	0.9300
C16—H16	0.9300	C38—H38	0.9300
C17—H17	0.9300		

C11—O1—C10	114.77 (11)	H18A—C18—H18B	108.4
C14—O3—C18	116.99 (12)	C24—C19—C20	118.91 (14)
C25—O4—C1	114.56 (11)	C24—C19—C18	120.55 (16)
C28—O6—C32	116.20 (11)	C20—C19—C18	120.46 (15)
C2—C1—C10	121.11 (14)	C21—C20—C19	120.28 (16)
C2—C1—O4	121.57 (15)	C21—C20—H20	119.9
C10—C1—O4	117.25 (13)	C19—C20—H20	119.9
C2—C1—O4	121.57 (15)	C22—C21—C20	120.24 (17)
C10—C1—O4	117.25 (13)	C22—C21—H21	119.9
C1—C2—C3	119.43 (15)	C20—C21—H21	119.9
C1—C2—H2	120.3	C21—C22—C23	119.68 (15)
C3—C2—H2	120.3	C21—C22—H22	120.2
C4—C3—C2	121.73 (15)	C23—C22—H22	120.2
C4—C3—C8	118.80 (14)	C22—C23—C24	120.11 (17)
C2—C3—C8	119.46 (14)	C22—C23—H23	119.9
C5—C4—C3	120.97 (16)	C24—C23—H23	119.9
C5—C4—H4	119.5	C19—C24—C23	120.77 (17)
C3—C4—H4	119.5	C19—C24—H24	119.6
C4—C5—C6	120.16 (15)	C23—C24—H24	119.6

C4—C5—H5	119.9	O5—C25—O4	121.75 (13)
C6—C5—H5	119.9	O5—C25—O4	121.75 (13)
C5—C6—C7	120.62 (15)	O5—C25—C26	126.09 (14)
C5—C6—H6	119.7	O4—C25—C26	112.15 (12)
C7—C6—H6	119.7	O4—C25—C26	112.15 (12)
C6—C7—C8	120.43 (16)	C31—C26—C27	121.00 (14)
C6—C7—H7	119.8	C31—C26—C25	117.65 (14)
C8—C7—H7	119.8	C27—C26—C25	121.28 (13)
C7—C8—C9	121.77 (15)	C28—C27—C26	119.14 (14)
C7—C8—C3	118.97 (14)	C28—C27—H27	120.4
C9—C8—C3	119.24 (14)	C26—C27—H27	120.4
C10—C9—C8	119.32 (15)	O6—C28—C27	124.34 (13)
C10—C9—H9	120.3	O6—C28—C29	115.74 (13)
C8—C9—H9	120.3	C27—C28—C29	119.91 (14)
C9—C10—O1	121.99 (15)	C30—C29—C28	120.33 (14)
C9—C10—O1	121.99 (15)	C30—C29—H29	119.8
C9—C10—C1	121.34 (14)	C28—C29—H29	119.8
O1—C10—C1	116.66 (13)	C29—C30—C31	120.67 (14)
O1—C10—C1	116.66 (13)	C29—C30—H30	119.7
O2—C11—O1	122.40 (14)	C31—C30—H30	119.7
O2—C11—O1	122.40 (14)	C26—C31—C30	118.91 (14)
O2—C11—C12	125.04 (13)	C26—C31—H31	120.5
O1—C11—C12	112.55 (12)	C30—C31—H31	120.5
O1—C11—C12	112.55 (12)	O6—C32—C33	108.43 (12)
C17—C12—C13	121.00 (14)	O6—C32—H32A	110.0
C17—C12—C11	122.14 (13)	C33—C32—H32A	110.0
C13—C12—C11	116.82 (13)	O6—C32—H32B	110.0
C14—C13—C12	119.04 (14)	C33—C32—H32B	110.0
C14—C13—H13	120.5	H32A—C32—H32B	108.4
C12—C13—H13	120.5	C34—C33—C38	119.16 (15)
O3—C14—C13	124.59 (14)	C34—C33—C32	120.50 (16)
O3—C14—C15	115.16 (13)	C38—C33—C32	120.30 (17)
C13—C14—C15	120.25 (14)	C33—C34—C35	120.74 (17)
C16—C15—C14	119.72 (15)	C33—C34—H34	119.6
C16—C15—H15	120.1	C35—C34—H34	119.6
C14—C15—H15	120.1	C36—C35—C34	119.64 (19)
C15—C16—C17	120.90 (15)	C36—C35—H35	120.2
C15—C16—H16	119.5	C34—C35—H35	120.2
C17—C16—H16	119.5	C35—C36—C37	119.75 (16)
C12—C17—C16	119.08 (14)	C35—C36—H36	120.1
C12—C17—H17	120.5	C37—C36—H36	120.1
C16—C17—H17	120.5	C38—C37—C36	120.58 (17)
O3—C18—C19	108.31 (12)	C38—C37—H37	119.7
O3—C18—H18A	110.0	C36—C37—H37	119.7
C19—C18—H18A	110.0	C37—C38—C33	120.11 (19)
O3—C18—H18B	110.0	C37—C38—H38	119.9
C19—C18—H18B	110.0	C33—C38—H38	119.9

C25—O4—C1—C2	−103.88 (16)	C13—C14—C15—C16	−1.3 (3)
C25—O4—C1—C10	79.14 (16)	C14—C15—C16—C17	1.4 (3)
O4—C1—C2—C3	−176.96 (12)	C13—C12—C17—C16	0.6 (3)
O4—C1—C2—C3	−176.96 (12)	C11—C12—C17—C16	178.47 (16)
C1—C2—C3—C4	176.92 (14)	C15—C16—C17—C12	−1.0 (3)
C1—C2—C3—C8	−2.3 (2)	C14—O3—C18—C19	172.84 (14)
C2—C3—C4—C5	−178.84 (14)	O3—C18—C19—C24	132.87 (16)
C3—C4—C5—C6	−1.4 (2)	O3—C18—C19—C20	−50.1 (2)
C5—C6—C7—C8	1.7 (2)	C24—C19—C20—C21	0.3 (2)
C6—C7—C8—C9	175.77 (13)	C18—C19—C20—C21	−176.77 (14)
C6—C7—C8—C3	−2.6 (2)	C19—C20—C21—C22	0.1 (2)
C4—C3—C8—C7	1.5 (2)	C20—C21—C22—C23	−0.4 (2)
C2—C3—C8—C7	−179.20 (13)	C21—C22—C23—C24	0.4 (3)
C4—C3—C8—C9	−176.86 (13)	C20—C19—C24—C23	−0.3 (2)
C2—C3—C8—C9	2.4 (2)	C18—C19—C24—C23	176.77 (15)
C7—C8—C9—C10	−178.40 (13)	C22—C23—C24—C19	−0.1 (3)
C8—C9—C10—O1	177.98 (12)	C1—O4—C25—O5	18.8 (2)
C8—C9—C10—O1	177.98 (12)	C1—O4—C25—O4	0 (22)
C8—C9—C10—C1	−2.4 (2)	C1—O4—C25—C26	−160.98 (13)
C11—O1—C10—C9	−85.49 (17)	O5—C25—C26—C31	13.0 (3)
C11—O1—C10—C1	94.88 (15)	O4—C25—C26—C31	−167.24 (14)
C2—C1—C10—C9	2.5 (2)	O4—C25—C26—C31	−167.24 (14)
O4—C1—C10—C9	179.53 (13)	O5—C25—C26—C27	−164.05 (16)
O4—C1—C10—C9	179.53 (13)	O4—C25—C26—C27	15.7 (2)
C2—C1—C10—O1	−177.83 (13)	O4—C25—C26—C27	15.7 (2)
O4—C1—C10—O1	−0.83 (18)	C31—C26—C27—C28	−0.6 (2)
O4—C1—C10—O1	−0.83 (18)	C25—C26—C27—C28	176.37 (15)
C2—C1—C10—O1	−177.83 (13)	C32—O6—C28—C27	−5.3 (2)
O4—C1—C10—O1	−0.83 (18)	C32—O6—C28—C29	173.76 (15)
O4—C1—C10—O1	−0.83 (18)	C26—C27—C28—O6	177.57 (15)
C10—O1—C11—O2	9.7 (2)	C26—C27—C28—C29	−1.5 (3)
C10—O1—C11—C12	−168.94 (14)	O6—C28—C29—C30	−176.56 (15)
O2—C11—C12—C17	176.16 (17)	C27—C28—C29—C30	2.6 (3)
O1—C11—C12—C17	−5.3 (2)	C28—C29—C30—C31	−1.6 (3)
O1—C11—C12—C17	−5.3 (2)	C27—C26—C31—C30	1.6 (3)
O2—C11—C12—C13	−5.9 (3)	C25—C26—C31—C30	−175.49 (15)
O1—C11—C12—C13	172.69 (14)	C29—C30—C31—C26	−0.5 (3)
O1—C11—C12—C13	172.69 (14)	C28—O6—C32—C33	−172.04 (14)
C17—C12—C13—C14	−0.6 (3)	O6—C32—C33—C34	85.55 (19)
C11—C12—C13—C14	−178.55 (15)	O6—C32—C33—C38	−96.72 (18)
C18—O3—C14—C13	2.9 (2)	C32—C33—C34—C35	176.88 (15)
C18—O3—C14—C15	−177.96 (15)	C34—C35—C36—C37	1.1 (3)
C12—C13—C14—O3	179.99 (15)	C36—C37—C38—C33	−1.0 (3)
C12—C13—C14—C15	0.9 (2)	C34—C33—C38—C37	1.6 (2)
O3—C14—C15—C16	179.51 (16)	C32—C33—C38—C37	−176.12 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···O5^{i}	0.93	2.52	3.258 (2)	136
C21—H21···O2^{ii}	0.93	2.49	3.378 (2)	160
C4—H4···*Cg*5^{iii}	0.93	2.60	3.4949 (19)	163
C16—H16···*Cg*2^{i}	0.93	2.95	3.6955 (18)	139
C17—H17···*Cg*1^{i}	0.93	2.90	3.7480 (17)	152
C17—H17···*Cg*7^{i}	0.93	2.91	3.6342 (17)	135
C18—H18*A*···*Cg*6^{ii}	0.97	2.66	3.5201 (18)	148
C30—H30···*Cg*1^{iv}	0.93	2.90	3.7078 (17)	146
C31—H31···*Cg*2^{iv}	0.93	2.69	3.5449 (17)	154
C31—H31···*Cg*7^{iv}	0.93	2.95	3.6130 (16)	130
C32—H32*A*···*Cg*4^{v}	0.97	2.82	3.5525 (18)	133
C15—H15···*Cg*6^{vi}	0.93	2.97	3.6860 (18)	135

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) −*x*+3/2, *y*+1/2, −*z*+1/2; (iii) −*x*+1, −*y*, −*z*; (iv) *x*−1, *y*, *z*; (v) −*x*+3/2, *y*−1/2, −*z*+1/2; (vi) −*x*+1/2, *y*+1/2, −*z*+1/2.