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Molecular and crystal structure of gossypol tetramethyl ether with an unknown solvate

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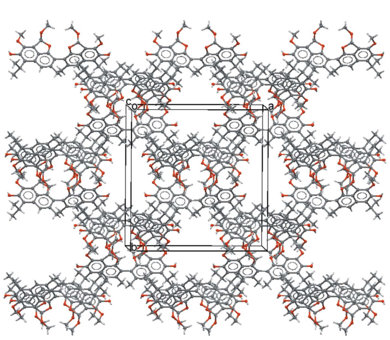
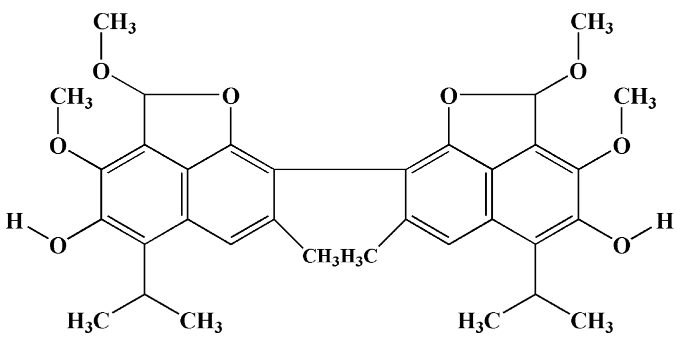
The title compound, $C_{34}H_{38}O_8$ (systematic name: 5,5′-diisopropyl-2,2′,3,3′-tetramethoxy-7,7′-dimethyl-2*H*,2′*H*-8,8′-bi[naphtho[1,8-*bc*]furan]-4,4′-diol), has been obtained from a gossypol solution in a mixture of dimethyl sulfate and methanol. The molecule is situated on a twofold rotation axis, so the asymmetric unit contains one half-molecule. In the molecule, the hydroxy groups are involved in intramolecular O—H···O hydrogen bonds, and the two naphthyl fragments are inclined each to other by 83.8 (1)°. In the crystal, weak C—H···O and C—H···π interactions consolidate the packing, which exhibits channels with an approximate diameter of 6 Å extending along the *c*-axis direction. These channels are filled with highly disordered solvent molecules, so their estimated scattering contribution was subtracted from the observed diffraction data using the SQUEEZE option in *PLATON* [Spek, A. L. (2015). *Acta Cryst*. C**71**, 9–18].



1. Chemical context

Gossypol [systematic name: 2,2′-bis(8-formyl-1,6,7-trihydroxyl-5-isopropyl-3-methylnaphthalene)] is an unique terpenoid found in *Gossypium* (cotton) and related species. Within plants, gossypol appears to act as a natural insecticide and fungicide (Adams *et al.*, 1960). Because of its antinutritive effect, gossypol limits the feeding of cottonseed and cottonseed meal to ruminant animals. However, the compound also has a wide range of biological actions, including anti-HIV, anticancer, and antifertility effects (Liang *et al.*, 1995; Dorsett *et al.*, 1975; Coutinho, 2002; Royer *et al.*, 1995). Gossypol is a surprisingly versatile host compound that forms inclusion complexes with a great variety of organic substances such as ketones, ethers, esters, organic and mineral acids, water, various benzyl compounds and chlorinated and brominated compounds. More than one hundred of these complexes with different guest molecules have been obtained and structurally characterized (Talipov *et al.*, 2002; 2003; 2007; Ibragimov *et al.*, 2004). A specific feature of gossypol is the existence of gossypol host–guest complexes in the form of polymorphic crystals. As a result of its comprehensive biological properties, there is current interest in the synthesis of new gossypol derivatives. Many derivatives have been reported, including ethers, acetates and Schiff bases with aldehydes (Talipov *et al.*, 2004; 2009; Tilyabaev *et al.*, 2009; Kenar, 2006). As first reported by Morris & Adams (1937), treatment with an alkali of a gossypol solution in a mixture of dimethyl sulfate and methanol, yields a white gossypol tetramethyl ether, the title compound.



2. Structural commentary

Gossypol can exist in one of the following tautomeric forms: aldehyde, quinoid and lactol (Adams *et al.*, 1960). In most solvents it is found in the aldehyde form. However, there are some reports that gossypol also exists in a pure lactol form (Reyes *et al.*, 1986) or as a dynamic equilibrium mixture of the aldehyde and lactol forms in some highly polar solvents (Kamaev *et al.*, 1979). In the structure described here, the title compound exists in the lactol form.

The crystallographically imposed symmetry of the title molecule is $C2$; the twofold axis is perpendicular to the C2—C2*A* bond [symmetry code (*A*): $-x, y, \frac{3}{2} - z$]. The symmetry of the molecule corresponds to symmetry of the crystal, the title compound molecule being situated on a twofold axis. An *ORTEP* diagram of the molecule showing the atom-numbering scheme is given in Fig. 1. The molecule consists of two fused ring systems, each containing a naphthalene ring system with a fused furan ring. The two napthyl bicycles of the molecule are nearly perpendicular and the dihedral angle between their least-squares planes is 83.8 (1)°. The furan ring is not completely planar, with atom C12 deviating from the C1/O1/C8/C9 plane by 0.225 (4) Å. The methoxy group at the C-7 position is almost coplanar with the plane of the naphthalene ring system; atomic deviations from this plane are 0.004 (3) for O3 and 0.163 (5) Å for C16. The methoxy group on the furan ring (C12-O2-C17H_3) and atom O1 are located on the same side of the host ring (C1–C4/C9/C10). The isopropyl groups are positioned with the ternary hydrogen atoms pointed outwards and away from the center of the molecule, the isopropyl groups bisect the extended naphthalene ring system plane.

There is an intramolecular O4—H4···O3 hydrogen bond (Table 1) which is similar to those observed previously in structures of gossypol and its Schiff bases. The values of the bond lengths and angles in the title molecule are within expected values. However, there are notable differences in the lengths of some of these bonds compared with typical values for gossypol structures. Compared with the relatively short C5—C6 aromatic ring bonds of gossypol molecules (1.36 Å), the corresponding bond in the title molecule is longer at 1.380 (3) Å. In addition, the C7—C8 and C8—C9 bonds in the title compound are shorter than those in gossypol by 0.03 and 0.06 Å, respectively. The shortest bond within these rings is the C1—C2 bond with a length of 1.359 (3) Å. In the furan ring, there are some differences in the lengths of some bonds compared with the values found in dianhydrogossypol. In the title molecule, the C1—O1 bond [1.374 (3) Å] is shorter than the O1—C12 bond [1.463 (3) Å].

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

Cg is the centroid of the C1–C4/C9/C10 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O3	0.67 (3)	2.17 (4)	2.586 (3)	122 (4)
C17—H17*A*···O1^{i}	0.96	2.71	3.286 (3)	119
C17—H17*C*···*Cg*^{ii}	0.96	2.77	3.551 (4)	139

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

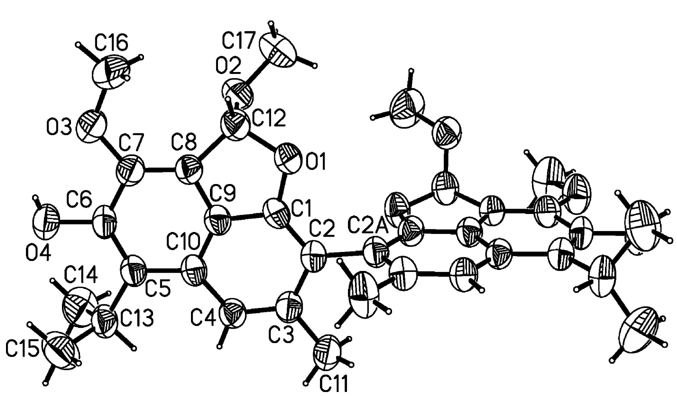


Figure 1
The molecular structure of the title compound showing the atomic numbering and 50% probability displacement ellipsoids. Unlabeled atoms are related to labeled ones by the symmetry operation (A) $-x, y, \frac{3}{2} - z$.

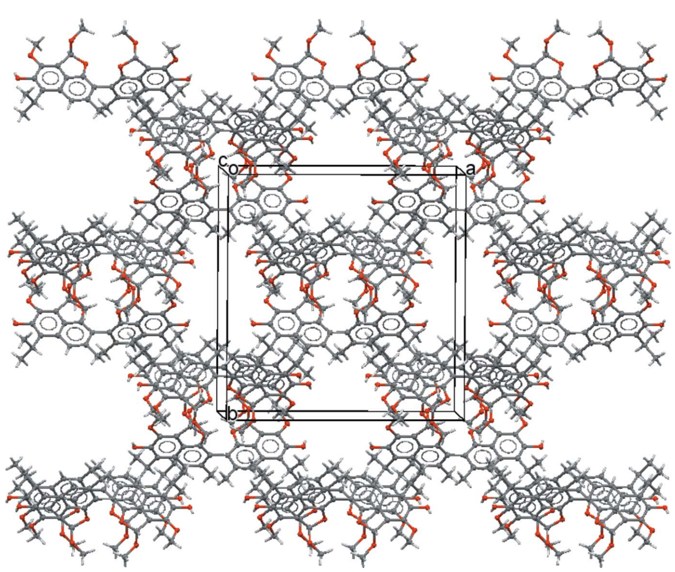


Figure 2
A portion of the crystal packing viewed approximately along the *c* axis.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{34}H_{38}O_8$
M_r	574.64
Crystal system, space group	Orthorhombic, *Pbcn*
Temperature (K)	293
a, *b*, *c* (Å)	19.7086 (5), 20.3099 (7), 8.8443 (4)
V (Å^3)	3540.2 (2)
Z	4
Radiation type	Cu *Kα*
μ (mm^{-1})	0.62
Crystal size (mm)	0.35 × 0.28 × 0.26
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Ruby
Absorption correction	Multi-scan (SCALE3 ABSPACK in *CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min}, T_{max}	0.914, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12345, 3340, 1826
R_{int}	0.049
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.613
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.162, 0.93
No. of reflections	3340
No. of parameters	200
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.17

Computer programs: *CrysAlis PRO* (Oxford Diffraction, 2009), *SHELXS97*, *SHELXL97* and *XP* in *SHELXTL* (Sheldrick, 2008).

3. Supramolecular features

The packing of the title molecules is shown in Fig. 2. Weak intermolecular C—H···O and C—H···π interactions (Table 1) consolidate the crystal packing, which exhibits channels with a diameter of approximately 6 Å extending along the *c*-axis direction. These channels are similar to the channels previously reported in a dianhydrogossypol crystal structure (Talipov *et al.*, 2009). In the present structure, for each unit cell, the channels provide a void volume of 672 Å^3 corresponding to 19% of the unit-cell volume. Highly disordered solvent molecules, most probably water molecules, occupy these voids in the crystal; their contribution to the scattering was removed with the SQUEEZE routine of the *PLATON* program (Spek, 2009, 2015).

4. Database survey

A search in the Cambridge Structural Database (Version 5.33, last update November 2013; Groom & Allen, 2014) indicated the presence of 191 entries for gossypol (137 entries) or gossypol derivatives. However, only four entries were found for fused-ring systems containing a naphthalene ring system with a fused furan ring. The dihedral angle between two fused ring systems in these structures is equal to 84.8 in TEYJEM (Ibragimov *et al.*, 1995), 111.8 in TEYJEN (Ibragimov *et al.*, 1995), 117.0 in YURMEE (Talipov *et al.*, 1999) and 119.1° in FOVKEG (Talipov *et al.*, 1999).

5. Synthesis and crystallization

Gossypol was obtained from the Experimental Plant of the Institute of Bioorganic Chemistry, Academy of Sciences of Uzbekistan where it was produced from by-products of the cottonseed oil industry. The title compound was synthesized following the known procedure (Morris & Adams, 1937). In order to prepare single crystals suitable for X-ray experiments, powdered material was dissolved in acetone (20 mg/1 ml) and stored for few days at room temperature under slow evaporation of the solution.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The H atom of the hydroxyl substituent was located in an electron density map and its coordinates were freely refined with U_{iso} = 1.5U_{eq}(O). C-bound H atoms were positioned geometrically and refined using a riding model, with d(C—H) = 0.93 Å and U_{iso} = 1.2U_{eq} (C) for aromatic, d(C—H) = 0.98 Å and U_{iso} = 1.2U_{eq} (C) for methine, d(C—H) = 0.96 Å and U_{iso} = 1.5U_{eq} (C) for methyl H atoms.

Acknowledgements

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supporting information

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Molecular and crystal structure of gossypol tetramethyl ether with an unknown solvate

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Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2009); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

5,5′-Diisopropyl-2,2′,3,3′-tetramethoxy-7,7′-dimethyl-2*H*,2′*H*-8,8′-bi[naphtho[1,8-*bc*]furan]-4,4′-diol

Crystal data

$C_{34}H_{38}O_8$
M_r = 574.64
Orthorhombic, *Pbcn*
a = 19.7086 (5) Å
b = 20.3099 (7) Å
c = 8.8443 (4) Å
V = 3540.2 (2) Å^3
Z = 4
F(000) = 1224
D_x = 1.078 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 2468 reflections
θ = 4.4–70.6°
μ = 0.62 mm^{-1}
T = 293 K
Prism, white
0.35 × 0.28 × 0.26 mm

Data collection

Oxford Diffraction Xcalibur Ruby diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.2576 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SCALE3 ABSPACK in *CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.914, T_{max} = 1.000
12345 measured reflections
3340 independent reflections
1826 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 71.0°, θ_{min} = 4.4°
$h = -23 \rightarrow 24$
$k = -22 \rightarrow 24$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.162
S = 0.93
3340 reflections
200 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0932P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}

$\Delta\rho_{min} = -0.17$ e Å^{-3}

Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00096 (17)

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}
O1	0.06600 (7)	0.08142 (8)	0.91284 (19)	0.0592 (5)
O2	0.12998 (9)	−0.01420 (8)	0.8665 (2)	0.0664 (5)
O3	0.29694 (8)	0.05026 (10)	0.9636 (2)	0.0735 (6)
O4	0.35682 (9)	0.13669 (12)	0.7953 (3)	0.0731 (6)
C1	0.08110 (10)	0.12972 (12)	0.8095 (3)	0.0487 (6)
C2	0.03731 (10)	0.17024 (12)	0.7358 (3)	0.0486 (6)
C3	0.06794 (11)	0.21591 (12)	0.6321 (3)	0.0521 (6)
C4	0.13705 (10)	0.22063 (12)	0.6150 (2)	0.0503 (6)
H4A	0.1545	0.2512	0.5472	0.060*
C5	0.25535 (11)	0.18076 (12)	0.6976 (2)	0.0498 (6)
C6	0.28733 (10)	0.13639 (12)	0.7919 (3)	0.0518 (6)
C7	0.25350 (11)	0.08978 (12)	0.8849 (3)	0.0532 (6)
C8	0.18412 (11)	0.08864 (11)	0.8835 (2)	0.0482 (6)
C9	0.15172 (10)	0.13466 (11)	0.7923 (2)	0.0465 (5)
C10	0.18240 (10)	0.18039 (12)	0.6973 (2)	0.0463 (6)
C11	0.02224 (12)	0.25791 (16)	0.5367 (3)	0.0750 (9)
H11B	0.0492	0.2873	0.4766	0.113*
H11C	−0.0073	0.2830	0.6009	0.113*
H11A	−0.0044	0.2303	0.4716	0.113*
C12	0.12826 (11)	0.04508 (13)	0.9467 (3)	0.0575 (7)
H12	0.1337	0.0379	1.0556	0.069*
C13	0.29419 (11)	0.22682 (14)	0.5945 (3)	0.0618 (7)
H13	0.2602	0.2522	0.5385	0.074*
C14	0.33502 (14)	0.18881 (18)	0.4773 (3)	0.0909 (10)
H14A	0.3052	0.1608	0.4204	0.136*
H14C	0.3686	0.1624	0.5273	0.136*
H14B	0.3570	0.2192	0.4102	0.136*
C15	0.33765 (15)	0.27649 (16)	0.6781 (4)	0.0922 (11)
H15B	0.3096	0.3020	0.7448	0.138*
H15A	0.3592	0.3052	0.6065	0.138*
H15C	0.3716	0.2538	0.7357	0.138*
C16	0.26946 (16)	0.00777 (18)	1.0766 (4)	0.1007 (12)

H16C	0.3057	−0.0154	1.1260	0.151*
H16A	0.2393	−0.0233	1.0301	0.151*
H16B	0.2450	0.0334	1.1496	0.151*
C17	0.08064 (15)	−0.06108 (16)	0.9165 (4)	0.0945 (11)
H17B	0.0860	−0.1014	0.8610	0.142*
H17A	0.0359	−0.0438	0.8997	0.142*
H17C	0.0869	−0.0695	1.0224	0.142*
H4	0.3667 (18)	0.1176 (19)	0.854 (4)	0.103 (15)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0420 (8)	0.0675 (11)	0.0681 (11)	0.0003 (8)	0.0123 (8)	0.0175 (9)
O2	0.0564 (10)	0.0584 (11)	0.0845 (13)	−0.0061 (8)	0.0122 (9)	0.0081 (10)
O3	0.0514 (10)	0.0864 (14)	0.0826 (13)	0.0050 (9)	−0.0047 (9)	0.0296 (11)
O4	0.0407 (10)	0.0939 (16)	0.0847 (15)	−0.0017 (9)	−0.0052 (9)	0.0252 (13)
C1	0.0415 (12)	0.0552 (14)	0.0493 (13)	−0.0065 (10)	0.0090 (10)	0.0020 (12)
C2	0.0375 (11)	0.0582 (14)	0.0501 (13)	0.0007 (11)	0.0027 (10)	−0.0023 (12)
C3	0.0425 (12)	0.0617 (15)	0.0519 (13)	0.0020 (11)	0.0005 (10)	0.0027 (12)
C4	0.0432 (12)	0.0590 (15)	0.0487 (13)	−0.0012 (11)	0.0035 (10)	0.0063 (11)
C5	0.0394 (11)	0.0610 (15)	0.0489 (13)	−0.0040 (10)	0.0019 (10)	0.0022 (12)
C6	0.0328 (11)	0.0673 (16)	0.0553 (13)	−0.0021 (11)	−0.0013 (10)	0.0006 (12)
C7	0.0454 (12)	0.0613 (16)	0.0530 (14)	0.0030 (11)	−0.0028 (11)	0.0063 (12)
C8	0.0412 (12)	0.0544 (14)	0.0491 (13)	0.0001 (10)	0.0029 (10)	0.0052 (11)
C9	0.0402 (11)	0.0545 (14)	0.0448 (12)	−0.0016 (10)	0.0042 (10)	0.0000 (11)
C10	0.0393 (11)	0.0569 (14)	0.0428 (12)	−0.0033 (10)	0.0037 (9)	−0.0035 (11)
C11	0.0463 (13)	0.094 (2)	0.085 (2)	0.0059 (14)	−0.0003 (13)	0.0254 (17)
C12	0.0486 (13)	0.0697 (17)	0.0543 (15)	0.0002 (12)	0.0060 (11)	0.0100 (13)
C13	0.0400 (12)	0.0816 (18)	0.0638 (16)	−0.0047 (12)	0.0038 (11)	0.0176 (14)
C14	0.0742 (19)	0.124 (3)	0.075 (2)	0.0012 (18)	0.0247 (16)	0.020 (2)
C15	0.086 (2)	0.097 (2)	0.094 (2)	−0.0331 (19)	−0.0049 (17)	0.024 (2)
C16	0.077 (2)	0.119 (3)	0.106 (2)	0.002 (2)	−0.0097 (18)	0.062 (2)
C17	0.084 (2)	0.081 (2)	0.119 (3)	−0.0242 (18)	0.0142 (19)	0.022 (2)

Geometric parameters (Å, º)

O1—C1	1.374 (3)	C8—C12	1.519 (3)
O1—C12	1.463 (3)	C9—C10	1.391 (3)
O2—C12	1.398 (3)	C11—H11B	0.9600
O2—C17	1.431 (3)	C11—H11C	0.9600
O3—C7	1.364 (3)	C11—H11A	0.9600
O3—C16	1.427 (3)	C12—H12	0.9800
O4—C6	1.370 (3)	C13—C15	1.516 (4)
O4—H4	0.67 (3)	C13—C14	1.523 (4)
C1—C2	1.359 (3)	C13—H13	0.9800
C1—C9	1.404 (3)	C14—H14A	0.9600
C2—C3	1.437 (3)	C14—H14C	0.9600
C2—C2^{i}	1.492 (4)	C14—H14B	0.9600

C3—C4	1.374 (3)	C15—H15B	0.9600
C3—C11	1.500 (3)	C15—H15A	0.9600
C4—C10	1.413 (3)	C15—H15C	0.9600
C4—H4A	0.9300	C16—H16C	0.9600
C5—C6	1.380 (3)	C16—H16A	0.9600
C5—C10	1.438 (3)	C16—H16B	0.9600
C5—C13	1.514 (3)	C17—H17B	0.9600
C6—C7	1.420 (3)	C17—H17A	0.9600
C7—C8	1.368 (3)	C17—H17C	0.9600
C8—C9	1.390 (3)		

C1—O1—C12	108.35 (16)	H11B—C11—H11A	109.5
C12—O2—C17	113.6 (2)	H11C—C11—H11A	109.5
C7—O3—C16	118.3 (2)	O2—C12—O1	110.5 (2)
C6—O4—H4	108 (3)	O2—C12—C8	107.31 (18)
C2—C1—O1	127.89 (19)	O1—C12—C8	103.82 (18)
C2—C1—C9	122.3 (2)	O2—C12—H12	111.6
O1—C1—C9	109.76 (19)	O1—C12—H12	111.6
C1—C2—C3	115.48 (19)	C8—C12—H12	111.6
C1—C2—C2^{i}	123.0 (2)	C5—C13—C15	113.8 (2)
C3—C2—C2^{i}	121.44 (19)	C5—C13—C14	111.3 (2)
C4—C3—C2	122.1 (2)	C15—C13—C14	111.8 (2)
C4—C3—C11	119.6 (2)	C5—C13—H13	106.5
C2—C3—C11	118.3 (2)	C15—C13—H13	106.5
C3—C4—C10	122.0 (2)	C14—C13—H13	106.5
C3—C4—H4A	119.0	C13—C14—H14A	109.5
C10—C4—H4A	119.0	C13—C14—H14C	109.5
C6—C5—C10	117.0 (2)	H14A—C14—H14C	109.5
C6—C5—C13	122.44 (19)	C13—C14—H14B	109.5
C10—C5—C13	120.5 (2)	H14A—C14—H14B	109.5
O4—C6—C5	117.8 (2)	H14C—C14—H14B	109.5
O4—C6—C7	117.3 (2)	C13—C15—H15B	109.5
C5—C6—C7	124.81 (19)	C13—C15—H15A	109.5
O3—C7—C8	128.5 (2)	H15B—C15—H15A	109.5
O3—C7—C6	113.13 (19)	C13—C15—H15C	109.5
C8—C7—C6	118.4 (2)	H15B—C15—H15C	109.5
C7—C8—C9	116.9 (2)	H15A—C15—H15C	109.5
C7—C8—C12	137.0 (2)	O3—C16—H16C	109.5
C9—C8—C12	105.79 (18)	O3—C16—H16A	109.5
C8—C9—C10	126.9 (2)	H16C—C16—H16A	109.5
C8—C9—C1	110.1 (2)	O3—C16—H16B	109.5
C10—C9—C1	123.0 (2)	H16C—C16—H16B	109.5
C9—C10—C4	114.98 (19)	H16A—C16—H16B	109.5
C9—C10—C5	115.9 (2)	O2—C17—H17B	109.5
C4—C10—C5	129.1 (2)	O2—C17—H17A	109.5
C3—C11—H11B	109.5	H17B—C17—H17A	109.5
C3—C11—H11C	109.5	O2—C17—H17C	109.5
H11B—C11—H11C	109.5	H17B—C17—H17C	109.5

C3—C11—H11A	109.5	H17A—C17—H17C	109.5
C12—O1—C1—C2	172.7 (2)	C7—C8—C9—C1	−176.7 (2)
C12—O1—C1—C9	−9.9 (3)	C12—C8—C9—C1	8.3 (3)
O1—C1—C2—C3	−179.4 (2)	C2—C1—C9—C8	178.3 (2)
C9—C1—C2—C3	3.6 (3)	O1—C1—C9—C8	0.8 (3)
O1—C1—C2—C2^{i}	3.4 (4)	C2—C1—C9—C10	−1.0 (4)
C9—C1—C2—C2^{i}	−173.7 (2)	O1—C1—C9—C10	−178.6 (2)
C1—C2—C3—C4	−3.4 (3)	C8—C9—C10—C4	178.9 (2)
C2^{i}—C2—C3—C4	173.9 (2)	C1—C9—C10—C4	−1.9 (3)
C1—C2—C3—C11	174.6 (2)	C8—C9—C10—C5	−1.7 (3)
C2^{i}—C2—C3—C11	−8.1 (4)	C1—C9—C10—C5	177.5 (2)
C2—C3—C4—C10	0.6 (4)	C3—C4—C10—C9	2.0 (3)
C11—C3—C4—C10	−177.4 (2)	C3—C4—C10—C5	−177.2 (2)
C10—C5—C6—O4	−179.0 (2)	C6—C5—C10—C9	−0.4 (3)
C13—C5—C6—O4	2.7 (4)	C13—C5—C10—C9	177.8 (2)
C10—C5—C6—C7	1.6 (4)	C6—C5—C10—C4	178.8 (2)
C13—C5—C6—C7	−176.6 (2)	C13—C5—C10—C4	−2.9 (4)
C16—O3—C7—C8	−10.0 (4)	C17—O2—C12—O1	−68.7 (3)
C16—O3—C7—C6	171.4 (2)	C17—O2—C12—C8	178.7 (2)
O4—C6—C7—O3	−1.3 (3)	C1—O1—C12—O2	−100.5 (2)
C5—C6—C7—O3	178.0 (2)	C1—O1—C12—C8	14.3 (2)
O4—C6—C7—C8	179.9 (2)	C7—C8—C12—O2	−70.0 (4)
C5—C6—C7—C8	−0.8 (4)	C9—C8—C12—O2	103.5 (2)
O3—C7—C8—C9	−179.8 (2)	C7—C8—C12—O1	172.9 (3)
C6—C7—C8—C9	−1.3 (4)	C9—C8—C12—O1	−13.6 (2)
O3—C7—C8—C12	−6.8 (5)	C6—C5—C13—C15	−64.3 (3)
C6—C7—C8—C12	171.7 (3)	C10—C5—C13—C15	117.5 (3)
C7—C8—C9—C10	2.6 (4)	C6—C5—C13—C14	63.1 (3)
C12—C8—C9—C10	−172.4 (2)	C10—C5—C13—C14	−115.1 (3)

Symmetry code: (i) $-x, y, -z+3/2$.

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C1–C4/C9/C10 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O3	0.67 (3)	2.17 (4)	2.586 (3)	122 (4)
C17—H17*A*···O1^{ii}	0.96	2.71	3.286 (3)	119
C17—H17*C*···*Cg*^{iii}	0.96	2.77	3.551 (4)	139

Symmetry codes: (ii) $-x, -y, -z+2$; (iii) $x, -y, z+1/2$.