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2-Butyl-5-pentylbenzene-1,3-diol

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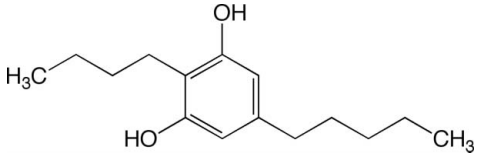
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.131; data-to-parameter ratio = 16.7.

In the title compound, $C_{15}H_{24}O_2$, a natural dialkylresorcinol commonly named stemphol, the molecules are linked into $C(6)$ and $C_2^2(4)$ chains and $R_4^4(16)$ rings by intermolecular O—H···O hydrogen bonds, creating molecular sheets parallel to the (010) plane. The alkyl chains are directed orthogonally away from these planes in almost complete extension.

Related literature

For general background, synthesis, biological activity and related structures, see: Achenbach & Kohl (1979); Andersen & Frisvad (2004); Marumo *et al.* (1985); Solfrizzo *et al.* (1994); Stodola *et al.* (1973). For structural discussion, see: Etter (1990); Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{15}H_{24}O_2$
M_r = 236.34
Monoclinic, $P2_1/c$
a = 4.654 (2) Å
b = 25.450 (5) Å
c = 12.790 (4) Å
β = 108.12 (1)°
V = 1439.8 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 293 K
0.50 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.881, T_{max} = 0.994
15852 measured reflections
2631 independent reflections
1703 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.131
S = 1.04
2631 reflections
158 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	1.96	2.767 (2)	167
O2—H2···O1^{ii}	0.82	1.95	2.750 (2)	165

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

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Nonius, 1999); cell refinement: *DENZO*; data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) and *CrystalBuilder* (Welter, 2006); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97* and *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2455).

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supporting information

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2-Butyl-5-pentylbenzene-1,3-diol

Juangjun Jumpathong, Pascal Retailleau, Muna Ali Abdalla, Jamal Ouazzani and Saisamorn Lumyong

S1. Comment

Stemphol was first isolated from *Stemphylium majusculum* (Stodola *et al.*, 1973) and its related compounds were reported with antimicrobial activities against fungus (*Mucor hiemalis*), yeast (*Schizosaccharomyces pombe*) and Gram positive bacteria (*Bacillus subtilis* and *Staphylococcus aureus*) (Achenbach & Kohl, 1979). Later on, stemphol was isolated from *Pleospora herbarum* and described as self-inhibitor (Marumo *et al.*, 1985). Five of eleven isolates of *Stemphylium botryosum* Wallr. from oilseed rape produced the phytotoxin, stemphol when cultured on rice (Solfrizzo *et al.*, 1994). *Stemphylium cf. lycopersici* was postharvest spoiler of fresh tomatoes and was first detected of stemphols in naturally contaminated tomatoes (Andersen & Frisvad, 2004). Screening investigation for polyketide and novel substance from new endophytic fungus led to the finding of stemphol from *Gaeumannomyces amomi* BCC4066 isolated from healthy pseudostem of *Alpinia malaccensis*, which was collected in the Suthep- Pui National Park, Chiang Mai, in the northern part of Thailand. This molecule has been reported for the first time in endophytic fungus and its X-ray structure (Fig. 1) is presented herein.

The two hydroxyl groups of the stemphol molecules lead to the formation of chains and rings through O—H···O hydrogen bonds (Table 1) with graph set motifs C(6), $C_2^2(4)$ and $R_4^4(16)$ (Etter, 1990; Bernstein *et al.*, 1995) all contained within the glide planes (Fig. 2). Both butyl and pentyl chains, directed in opposite directions and running up and down with respect to the resorcinol mean plane, adopt essentially fully extended conformations, but with 12.7 (2)° of deviation between their mean planes defined by C1–C7/C10 and C4–C12/C15 respectively (Fig. 3). The dihedral angles they form with respect to the (010) plane are 84.3 (2)° and 73.5 (2)° respectively.

S2. Experimental

Gaeumannomyces amomi BCC4066 was cultivated in 20 l of liquid culture and incubated for 21 d at room temperature (20 °C). Liquid culture was filtrated and extracted separately. The residue (5.0 g) obtained after evaporation of solvent was subjected to column chromatography over sephadex to afford 30 mg of Stemphol. Colourless needle-shaped crystals were obtained by re-crystallization from 20% EtOAc in Heptane (m.p. 91 °C).

S3. Refinement

All H atoms were located in difference maps but were treated as riding on their parent atoms, with O—H = 0.82 Å, and C—H distances in the range 0.93–0.97 Å and with U_{iso}(H) = 1.2U_{eq}(carrier) [1.5 for methyl H atoms].

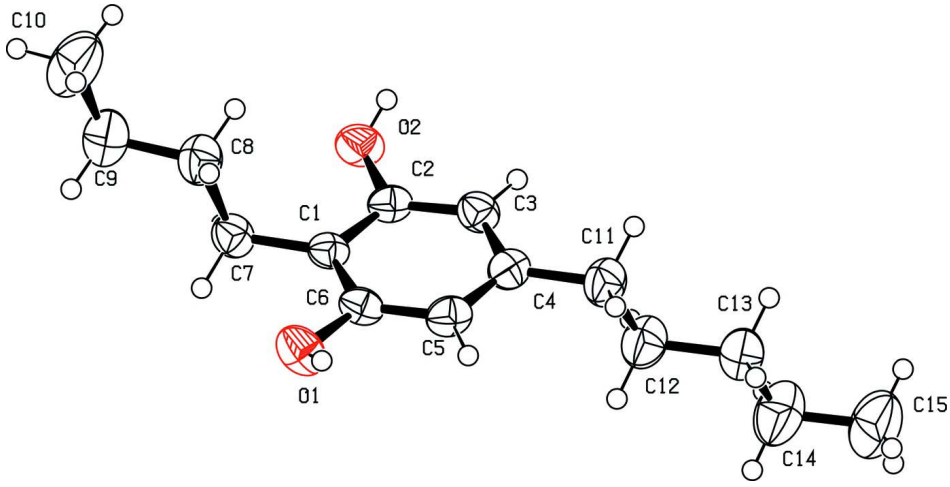

Figure 1

Molecular view of the compound with the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

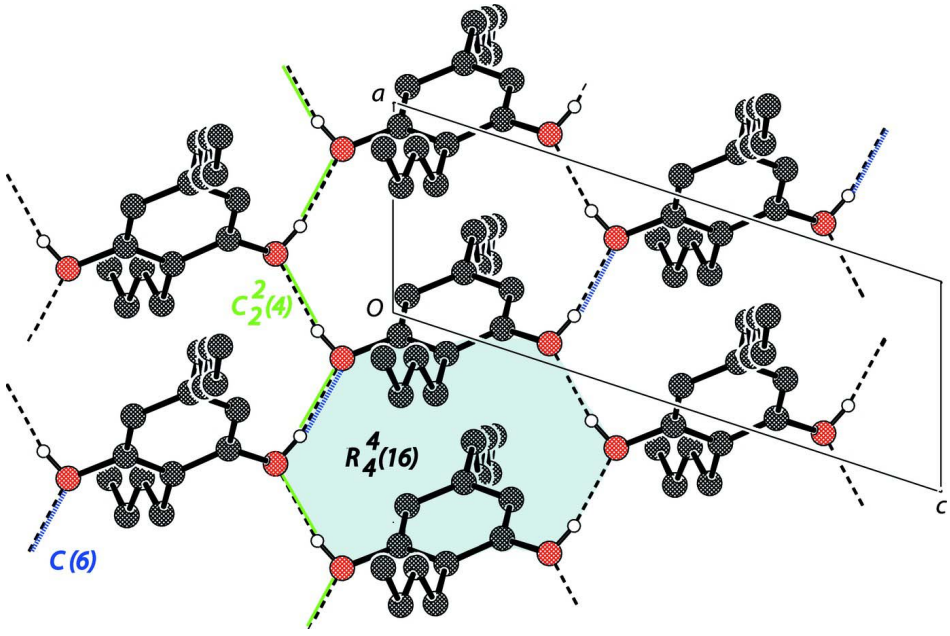

Figure 2

The crystal packing of the title molecule, viewed down the *b* axis, showing the molecules connected by O—H···O hydrogen bonds in dotted lines. H atoms not involved in hydrogen bondings have been omitted for clarity.

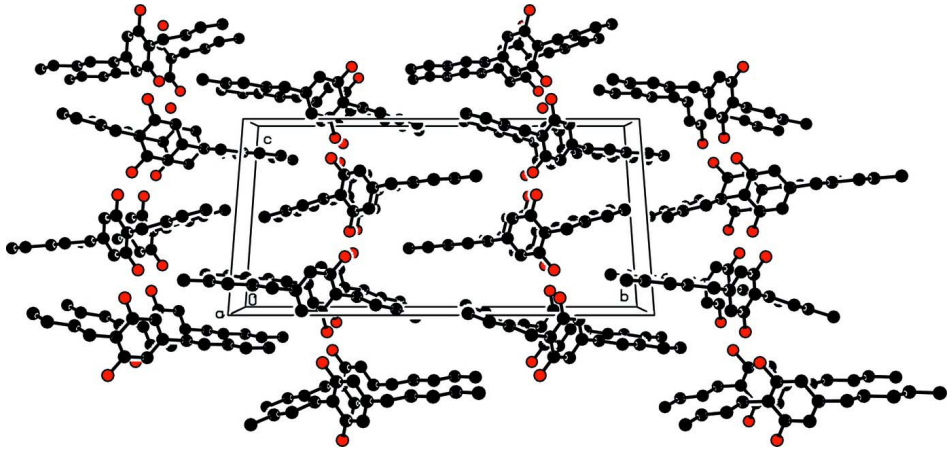

Figure 3

The crystal packing of the title molecule, viewed down the *a* axis, showing the segregration between alkyl chains and resorcinol moieties; H atoms are omitted for clarity.

2-Butyl-5-pentylbenzene-1,3-diol

Crystal data

$C_{15}H_{24}O_2$
$M_r = 236.34$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 4.654$ (2) Å
$b = 25.450$ (5) Å
$c = 12.790$ (4) Å
$\beta = 108.12$ (1)°
$V = 1439.8$ (8) Å^3
$Z = 4$
$F(000) = 520$
$D_x = 1.090$ Mg m^{-3}
Melting point: 364 K
Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
Cell parameters from 2700 reflections
$\theta = 0.4$–$25.4°$
$\mu = 0.07$ mm^{-1}
$T = 293$ K
Needle, colourless
0.50 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.881$, $T_{max} = 0.994$
15852 measured reflections
2631 independent reflections
1703 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.3°$
$h = -5 \rightarrow 5$
$k = -30 \rightarrow 30$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.131$
$S = 1.04$
2631 reflections
158 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0625P)^2 + 0.1805P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ 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}
O1	0.0951 (3)	0.27129 (5)	0.28490 (10)	0.0555 (4)
H1	0.2648	0.2719	0.3299	0.083*
O2	−0.2986 (2)	0.22721 (5)	−0.09324 (9)	0.0517 (4)
H2	−0.2074	0.2253	−0.1386	0.077*
C1	−0.1145 (3)	0.24879 (6)	0.09661 (13)	0.0358 (4)
C2	−0.1012 (3)	0.21631 (7)	0.01107 (13)	0.0383 (4)
C3	0.0994 (4)	0.17495 (7)	0.02531 (14)	0.0428 (4)
H3	0.1000	0.1542	−0.0345	0.051*
C4	0.3006 (3)	0.16426 (7)	0.12888 (14)	0.0414 (4)
C5	0.2925 (3)	0.19620 (7)	0.21530 (14)	0.0432 (5)
H5	0.4233	0.1896	0.2855	0.052*
C6	0.0935 (3)	0.23774 (7)	0.19898 (13)	0.0394 (4)
C7	−0.3211 (4)	0.29564 (7)	0.07705 (15)	0.0454 (5)
H7A	−0.3674	0.3038	0.1441	0.054*
H7B	−0.5092	0.2871	0.0207	0.054*
C8	−0.1801 (4)	0.34346 (7)	0.04135 (17)	0.0557 (5)
H8A	0.0020	0.3528	0.0999	0.067*
H8B	−0.1210	0.3341	−0.0226	0.067*
C9	−0.3832 (5)	0.39095 (8)	0.0136 (2)	0.0756 (7)
H9A	−0.5625	0.3822	−0.0468	0.091*
H9B	−0.4472	0.3999	0.0766	0.091*
C10	−0.2332 (6)	0.43810 (9)	−0.0182 (3)	0.1069 (10)
H10A	−0.0721	0.4500	0.0445	0.160*
H10B	−0.3790	0.4657	−0.0430	0.160*
H10C	−0.1529	0.4286	−0.0762	0.160*
C11	0.5085 (4)	0.11754 (7)	0.14655 (16)	0.0523 (5)
H11A	0.6758	0.1228	0.2134	0.063*
H11B	0.5910	0.1151	0.0858	0.063*
C12	0.3515 (4)	0.06650 (7)	0.15543 (18)	0.0604 (6)
H12A	0.1767	0.0628	0.0905	0.073*
H12B	0.2783	0.0687	0.2185	0.073*
C13	0.5435 (4)	0.01760 (7)	0.16697 (17)	0.0594 (5)
H13A	0.6171	0.0153	0.1040	0.071*
H13B	0.7178	0.0211	0.2322	0.071*
C14	0.3833 (5)	−0.03251 (8)	0.1752 (2)	0.0848 (8)

H14A	0.3137	−0.0304	0.2391	0.102*
H14B	0.2060	−0.0354	0.1109	0.102*
C15	0.5672 (6)	−0.08146 (9)	0.1840 (2)	0.0970 (9)
H15A	0.6319	−0.0848	0.1200	0.145*
H15B	0.4468	−0.1114	0.1892	0.145*
H15C	0.7409	−0.0796	0.2485	0.145*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0453 (7)	0.0768 (9)	0.0399 (8)	0.0016 (7)	0.0069 (6)	−0.0146 (7)
O2	0.0446 (7)	0.0744 (9)	0.0314 (7)	0.0080 (6)	0.0052 (5)	0.0047 (7)
C1	0.0292 (8)	0.0443 (10)	0.0339 (9)	−0.0028 (7)	0.0099 (7)	0.0024 (8)
C2	0.0316 (8)	0.0488 (10)	0.0311 (9)	−0.0041 (8)	0.0046 (7)	0.0039 (8)
C3	0.0432 (9)	0.0484 (11)	0.0367 (10)	0.0004 (9)	0.0120 (8)	−0.0031 (8)
C4	0.0345 (8)	0.0442 (10)	0.0442 (11)	−0.0015 (8)	0.0104 (8)	0.0053 (8)
C5	0.0333 (9)	0.0552 (11)	0.0350 (10)	−0.0007 (8)	0.0018 (7)	0.0063 (9)
C6	0.0342 (9)	0.0511 (11)	0.0325 (10)	−0.0050 (8)	0.0101 (7)	−0.0029 (8)
C7	0.0369 (9)	0.0538 (11)	0.0450 (11)	0.0028 (8)	0.0119 (8)	−0.0009 (9)
C8	0.0491 (10)	0.0527 (12)	0.0640 (13)	0.0043 (9)	0.0156 (9)	0.0016 (10)
C9	0.0815 (15)	0.0596 (14)	0.0889 (17)	0.0159 (12)	0.0310 (13)	0.0062 (13)
C10	0.131 (2)	0.0567 (16)	0.138 (3)	0.0108 (15)	0.050 (2)	0.0224 (16)
C11	0.0440 (10)	0.0511 (11)	0.0592 (12)	0.0069 (9)	0.0124 (8)	0.0052 (10)
C12	0.0517 (11)	0.0524 (12)	0.0753 (14)	0.0037 (9)	0.0168 (10)	0.0068 (11)
C13	0.0604 (12)	0.0521 (12)	0.0638 (14)	0.0075 (10)	0.0167 (10)	0.0006 (10)
C14	0.0828 (16)	0.0543 (14)	0.113 (2)	0.0030 (12)	0.0242 (15)	0.0058 (13)
C15	0.118 (2)	0.0560 (15)	0.113 (2)	0.0078 (14)	0.0293 (17)	−0.0002 (14)

Geometric parameters (Å, º)

O1—C6	1.3899 (19)	C9—H9A	0.9700
O1—H1	0.8200	C9—H9B	0.9700
O2—C2	1.3924 (18)	C10—H10A	0.9600
O2—H2	0.8200	C10—H10B	0.9600
C1—C2	1.388 (2)	C10—H10C	0.9600
C1—C6	1.394 (2)	C11—C12	1.512 (3)
C1—C7	1.503 (2)	C11—H11A	0.9700
C2—C3	1.381 (2)	C11—H11B	0.9700
C3—C4	1.391 (2)	C12—C13	1.512 (3)
C3—H3	0.9300	C12—H12A	0.9700
C4—C5	1.382 (2)	C12—H12B	0.9700
C4—C11	1.505 (2)	C13—C14	1.497 (3)
C5—C6	1.378 (2)	C13—H13A	0.9700
C5—H5	0.9300	C13—H13B	0.9700
C7—C8	1.519 (3)	C14—C15	1.496 (3)
C7—H7A	0.9700	C14—H14A	0.9700
C7—H7B	0.9700	C14—H14B	0.9700
C8—C9	1.507 (3)	C15—H15A	0.9600

C8—H8A	0.9700	C15—H15B	0.9600
C8—H8B	0.9700	C15—H15C	0.9600
C9—C10	1.506 (3)		

C6—O1—H1	109.5	H9A—C9—H9B	107.8
C2—O2—H2	109.5	C9—C10—H10A	109.5
C2—C1—C6	115.59 (15)	C9—C10—H10B	109.5
C2—C1—C7	121.63 (14)	H10A—C10—H10B	109.5
C6—C1—C7	122.55 (15)	C9—C10—H10C	109.5
C3—C2—C1	122.97 (15)	H10A—C10—H10C	109.5
C3—C2—O2	119.73 (15)	H10B—C10—H10C	109.5
C1—C2—O2	117.29 (14)	C4—C11—C12	112.77 (14)
C2—C3—C4	120.13 (16)	C4—C11—H11A	109.0
C2—C3—H3	119.9	C12—C11—H11A	109.0
C4—C3—H3	119.9	C4—C11—H11B	109.0
C5—C4—C3	117.98 (15)	C12—C11—H11B	109.0
C5—C4—C11	121.38 (16)	H11A—C11—H11B	107.8
C3—C4—C11	120.57 (17)	C11—C12—C13	115.46 (16)
C6—C5—C4	120.95 (15)	C11—C12—H12A	108.4
C6—C5—H5	119.5	C13—C12—H12A	108.4
C4—C5—H5	119.5	C11—C12—H12B	108.4
C5—C6—O1	120.90 (14)	C13—C12—H12B	108.4
C5—C6—C1	122.35 (16)	H12A—C12—H12B	107.5
O1—C6—C1	116.74 (14)	C14—C13—C12	114.58 (17)
C1—C7—C8	111.85 (14)	C14—C13—H13A	108.6
C1—C7—H7A	109.2	C12—C13—H13A	108.6
C8—C7—H7A	109.2	C14—C13—H13B	108.6
C1—C7—H7B	109.2	C12—C13—H13B	108.6
C8—C7—H7B	109.2	H13A—C13—H13B	107.6
H7A—C7—H7B	107.9	C15—C14—C13	115.4 (2)
C9—C8—C7	114.57 (16)	C15—C14—H14A	108.4
C9—C8—H8A	108.6	C13—C14—H14A	108.4
C7—C8—H8A	108.6	C15—C14—H14B	108.4
C9—C8—H8B	108.6	C13—C14—H14B	108.4
C7—C8—H8B	108.6	H14A—C14—H14B	107.5
H8A—C8—H8B	107.6	C14—C15—H15A	109.5
C10—C9—C8	113.16 (18)	C14—C15—H15B	109.5
C10—C9—H9A	108.9	H15A—C15—H15B	109.5
C8—C9—H9A	108.9	C14—C15—H15C	109.5
C10—C9—H9B	108.9	H15A—C15—H15C	109.5
C8—C9—H9B	108.9	H15B—C15—H15C	109.5

C6—C1—C2—C3	1.2 (2)	C7—C1—C6—C5	−176.77 (15)
C7—C1—C2—C3	175.85 (15)	C2—C1—C6—O1	176.89 (14)
C6—C1—C2—O2	−177.88 (13)	C7—C1—C6—O1	2.3 (2)
C7—C1—C2—O2	−3.2 (2)	C2—C1—C7—C8	−83.9 (2)
C1—C2—C3—C4	−0.1 (2)	C6—C1—C7—C8	90.4 (2)
O2—C2—C3—C4	179.01 (14)	C1—C7—C8—C9	176.41 (17)

C2—C3—C4—C5	−0.2 (2)	C7—C8—C9—C10	178.26 (19)
C2—C3—C4—C11	176.65 (15)	C5—C4—C11—C12	98.0 (2)
C3—C4—C5—C6	−0.8 (2)	C3—C4—C11—C12	−78.7 (2)
C11—C4—C5—C6	−177.58 (15)	C4—C11—C12—C13	176.76 (17)
C4—C5—C6—O1	−177.00 (15)	C11—C12—C13—C14	−179.8 (2)
C4—C5—C6—C1	2.0 (2)	C12—C13—C14—C15	178.6 (2)
C2—C1—C6—C5	−2.2 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82	1.96	2.767 (2)	167
O2—H2···O1^{ii}	0.82	1.95	2.750 (2)	165

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