Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

trans-5,6-Diphenylperhydropyran-2,4-dione

Laura C. de Souza, Dennis de O. Imbroisi, Carlos A. De Simone,* Mariano A. Pereira and Valéria R. S. Malta

Instituto de Química e Biotecnologia, Universidade Federal de Alagoas, 57072-970 Maceió, AL, Brazil
Correspondence e-mail: cas@qui.ufal.br

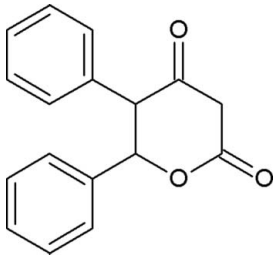
Received 21 November 2008; accepted 2 January 2009

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.050; wR factor = 0.131; data-to-parameter ratio = 17.2.

In the title compound, $C_{17}H_{14}O_3$, the pyran ring adopts a boat conformation and the dihedral angle between the aromatic ring planes is 59.1 (1)°. In the crystal structure intermolecular C—H···O hydrogen bonds and C—H···π interactions link the molecules.

Related literature

For general background, see: Yen & Chen (1995); Soler-Rivas *et al.* (2000). For related structures and biological activity, see: Brand-William *et al.* (1995); Sánchez-Moreno *et al.* (1998); Souza *et al.* (2004). For the synthesis, see: Souza (2008). For geometric analysis, see: Cremer & Pople (1975). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{17}H_{14}O_3$
M_r = 266.28
Monoclinic, $P2_1/c$
a = 8.9940 (2) Å
b = 8.2310 (4) Å
c = 18.9040 (8) Å
β = 101.412 (2)°
V = 1371.79 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 K
0.30 × 0.30 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
5298 measured reflections
3113 independent reflections
2459 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.131
S = 1.05
3113 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O2^{i}	0.98	2.44	3.380 (2)	161
C17—H17···O3^{ii}	0.93	2.46	3.351 (3)	160
C3—H3B···$Cg1^{i}$	0.97	2.97	3.681 (2)	131
C5—H5···$Cg2^{iii}$	0.98	2.96	3.830 (2)	149

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x, -y-\frac{1}{2}, z+\frac{1}{2}$; (iii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$. $Cg1$ and $Cg2$ are the centroids of the C7–C12 and C13–C18 rings, respectively.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work has received partial support from CNPq, CAPES, FAPEAL, IM-INOFAR and FINEP.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2110).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Brand-William, W., Cuvelier, M. E. & Berset, C. (1995). *Food Sci. Technol.* **28**, 25–30.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326, New York: Academic Press.
Sánchez-Moreno, C., Larrauri, J. A. & Saura-Calixto, F. J. (1998). *Sci. Food. Agric.* **76**, 270–276.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Soler-Rivas, C., Espin, J. C. & Withhers, H. J. (2000). *Phytochem. Anal.* **11**, 330–338.
Souza, L. C. (2008). PhD thesis, Instituto de Química e Biotecnologia, University of Alagoas, Brazil.
Souza, L. C., Araújo, S. M. S. & Imbroisi, D. O. (2004). *Bioorg. Med. Chem. Lett.* **14**, 5859–5861.
Yen, G. C. & Chen, H. Y. J. (1995). *Agric. Food Chem.* **43**, 27–32.

supporting information

Acta Cryst. (2009). E**65**, o250 [doi:10.1107/S1600536809000087]

trans-5,6-Diphenylperhydropyran-2,4-dione

Laura C. de Souza, Dennis de O. Imbroisi, Carlos A. De Simone, Mariano A. Pereira and Valéria R. S. Malta

S1. Comment

The free radicals generated in bioorganic redoxi processes induce oxidative damage in various components of the cells (*e.g.*, lipids, proteins and nucleic acids) and their play a significant role in the development of life-limiting chronic diseases such as cancer, hypertension, arteriosclerosis, rheumatism, cataracts and other (Yen & Chen, 1995; Soler-Rivas *et al.*,2000). The dihydropyran-2,4-diones exhibit structural features present bin many biologically active natural products possessing important pharmacological activities (Brand-William *et al.*,1995; Sánchez-Moreno *et al.*,1998). As part of our continuing studies aimed at ascertaining the biological activity of this class, the title compound was synthetized (Souza, 2008) and its antioxidant activity analyzed *in vitro*, by measuring the decrease in absorbance at 515 nm that occurred when the 2,2-diphenyl-1-picryl-hydrazyl radical (DPPH) was reduced by the antioxidant. The spectrophotometric assay was used to determine the radical scavenging activity (Souza *et al.*,2004).

The *ORTEP-3* (Farrugia, 1997) representation of the title compound (5,6-DPDP) is showing in (Fig. 1). Bond lengths and angles are in good agreement with the expected values reported in the literature (Allen *et al.*, 1987). The pirane ring adopts a boat conformation and the calculated puckering parameters are: q_2 = 0.624 (1) Å, q_3 = 0.121 (1) Å, Q_T = 0.636 (1) Å, θ = 79.0 (1)° and φ = 287.5 (1)° (Cremer & Pople, 1975). The dihedral angle between planes passing through atoms C7—C12 and C13—C18 of the aromatic rings is 59.1 (1) °. In the crystal packing, molecules interact through two intermolecular C–H···O hydrogen bonds and two C—H···.π interactions, Fig. 2 and Table 1.

S2. Experimental

The *trans*-5, 6-diphenyltetradehydropyran-2,4-dione has showed similar antioxidant activity at the positive control, the synthetic antioxidant BHT (2,6-di-*tert*-butyl-4-methylphenol) used as food conserving. The reduction percentage after 60 minutes to a solution of 20 nM of sample were 88% to 5,6-DPDP and 82% to BHT (Souza, 2008).The 5,6-DPDP was synthesized in one pot by preparation of the dianion of the ethyl 3-oxo-4-phenylbutanoate (NaH, n-butillithium, THF, -10° C), and alkylation reaction with benzaldehyde followed by ester hydrolysis (NaOH, H_2O, 12 h, RT) and lactonization in acidic medium (HCl, H_2O, 2 h, 0°C). The compound was purified by silica gel chromatography and the crystals for *x*-ray diffraction studies were grown by slow evaporation from a $CHCl_3$ solution.

S3. Refinement

H atoms were located on stereochemical grounds and refined with fixed geometry, each riding on a carrier atom, with C —H = [0.93 - 0.98] Å and anisotropic displacement parameter amounting to 1.5 (for Methyl-H atoms) and 1.2 (for the other H atoms) times the value of the equivalent isotropic displacement parameter of the which they are attached.The maximum and minimum residual electron density peaks were located 0.73 and 0.74 Å, from the C5 and H15 atoms respectively.

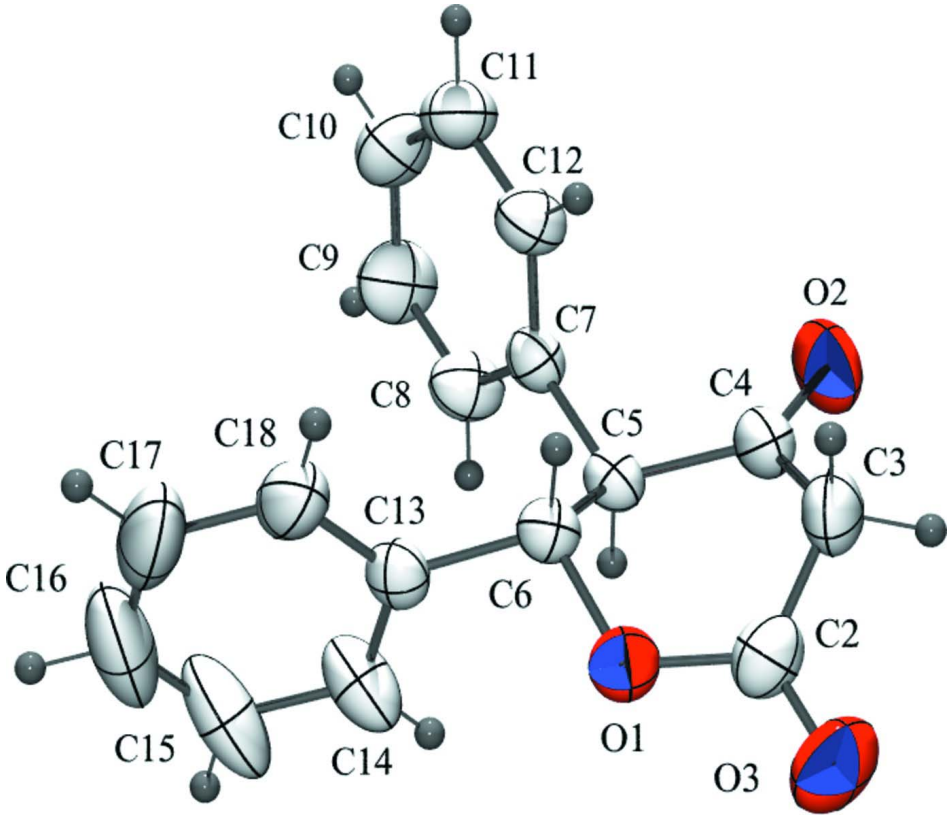


Figure 1

Projection of $C_{17}H_{14}O_3$, showing the atom labelling with 50% probability displacement.

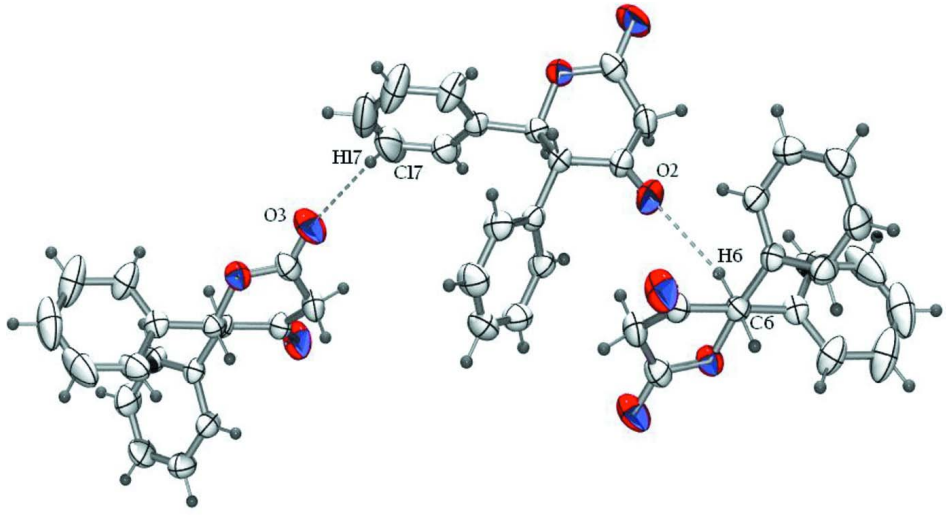


Figure 2

Hydrogen interactions.

trans-5,6-Diphenylperhydropyran-2,4-dione

Crystal data

$C_{17}H_{14}O_3$
$M_r = 266.28$
Monoclinic, $P2_1/c$
Hall symbol: -P2ybc
$a = 8.9940\ (2)$ Å
$b = 8.2310\ (4)$ Å
$c = 18.9040\ (8)$ Å
$\beta = 101.412\ (2)°$
$V = 1371.79\ (9)$ Å^3
$Z = 4$
$F(000) = 560$
$D_x = 1.289$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2880 reflections
$\theta = 1.0–27.5°$
$\mu = 0.09$ mm^{-1}
$T = 295$ K
Prism, yellow
0.30 × 0.30 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: Enraf Nonius FR590
Horizonally mounted graphite crystal monochromator
Detector resolution: 9 pixels mm^{-1}
CCD rotation images, thick slices scans
5298 measured reflections
3113 independent reflections
2459 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.3°$
$h = -11\rightarrow11$
$k = -9\rightarrow10$
$l = -24\rightarrow24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.131$
$S = 1.05$
3113 reflections
181 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.3831P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C2	0.69397 (19)	−0.0595 (2)	0.63794 (8)	0.0521 (4)
C3	0.84834 (19)	0.0134 (2)	0.65634 (8)	0.0560 (4)
H3A	0.8717	0.0620	0.6131	0.067*
H3B	0.9211	−0.0730	0.6712	0.067*
C4	0.86937 (16)	0.1403 (2)	0.71484 (8)	0.0476 (4)

C5	0.76480 (14)	0.12492 (17)	0.76852 (7)	0.0383 (3)
H5	0.6771	0.1953	0.7515	0.046*
C6	0.70604 (15)	−0.05049 (17)	0.76642 (7)	0.0385 (3)
H6	0.7925	−0.1246	0.7790	0.046*
C7	0.83633 (14)	0.17992 (16)	0.84390 (7)	0.0385 (3)
C8	0.76586 (17)	0.2946 (2)	0.87924 (9)	0.0520 (4)
H8	0.6745	0.3402	0.8562	0.062*
C9	0.8303 (2)	0.3421 (2)	0.94867 (10)	0.0652 (5)
H9	0.7817	0.4189	0.9722	0.078*
C10	0.9657 (2)	0.2763 (2)	0.98306 (9)	0.0631 (5)
H10	1.0094	0.3097	1.0295	0.076*
C11	1.03660 (18)	0.1616 (2)	0.94891 (9)	0.0565 (4)
H11	1.1281	0.1166	0.9723	0.068*
C12	0.97190 (16)	0.11267 (19)	0.87959 (8)	0.0477 (4)
H12	1.0198	0.0340	0.8567	0.057*
C13	0.59831 (15)	−0.07985 (17)	0.81644 (7)	0.0412 (3)
C14	0.46566 (19)	0.0086 (2)	0.80971 (12)	0.0644 (5)
H14	0.4401	0.0834	0.7724	0.077*
C15	0.3711 (3)	−0.0137 (3)	0.85803 (16)	0.0935 (8)
H15	0.2827	0.0473	0.8535	0.112*
C16	0.4061 (3)	−0.1242 (4)	0.91217 (15)	0.1019 (10)
H16	0.3420	−0.1378	0.9447	0.122*
C17	0.5352 (3)	−0.2153 (4)	0.91893 (11)	0.0967 (9)
H17	0.5583	−0.2915	0.9558	0.116*
C18	0.6323 (2)	−0.1938 (2)	0.87041 (9)	0.0643 (5)
H18	0.7197	−0.2564	0.8746	0.077*
O1	0.62369 (12)	−0.08786 (13)	0.69346 (5)	0.0487 (3)
O2	0.96257 (15)	0.24604 (19)	0.71798 (7)	0.0767 (4)
O3	0.63076 (17)	−0.0935 (2)	0.57795 (6)	0.0792 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0651 (10)	0.0484 (9)	0.0431 (8)	0.0015 (7)	0.0113 (7)	−0.0051 (7)
C3	0.0617 (9)	0.0655 (10)	0.0462 (8)	−0.0027 (8)	0.0239 (7)	−0.0015 (7)
C4	0.0423 (7)	0.0550 (9)	0.0467 (8)	−0.0069 (7)	0.0113 (6)	0.0061 (7)
C5	0.0347 (6)	0.0387 (7)	0.0422 (7)	−0.0028 (5)	0.0091 (5)	0.0005 (6)
C6	0.0368 (6)	0.0390 (7)	0.0393 (7)	−0.0005 (5)	0.0065 (5)	−0.0010 (5)
C7	0.0355 (6)	0.0375 (7)	0.0436 (7)	−0.0054 (5)	0.0105 (5)	−0.0013 (6)
C8	0.0456 (8)	0.0520 (9)	0.0578 (9)	0.0054 (7)	0.0089 (7)	−0.0089 (7)
C9	0.0683 (11)	0.0677 (11)	0.0606 (10)	0.0065 (9)	0.0158 (8)	−0.0212 (9)
C10	0.0674 (11)	0.0741 (12)	0.0456 (9)	−0.0053 (9)	0.0054 (7)	−0.0149 (8)
C11	0.0462 (8)	0.0640 (10)	0.0558 (9)	0.0005 (7)	0.0015 (7)	−0.0007 (8)
C12	0.0400 (7)	0.0506 (8)	0.0524 (8)	0.0012 (6)	0.0091 (6)	−0.0066 (7)
C13	0.0426 (7)	0.0381 (7)	0.0435 (7)	−0.0100 (5)	0.0100 (6)	−0.0046 (6)
C14	0.0544 (9)	0.0477 (9)	0.0998 (14)	−0.0010 (7)	0.0365 (9)	0.0020 (9)
C15	0.0823 (14)	0.0728 (14)	0.147 (2)	−0.0182 (11)	0.0759 (15)	−0.0221 (15)
C16	0.113 (2)	0.118 (2)	0.0933 (17)	−0.0592 (18)	0.0672 (16)	−0.0339 (16)

C17	0.1149 (19)	0.122 (2)	0.0517 (11)	−0.0542 (18)	0.0137 (12)	0.0191 (12)
C18	0.0620 (10)	0.0742 (12)	0.0529 (9)	−0.0146 (9)	0.0023 (8)	0.0170 (9)
O1	0.0522 (6)	0.0502 (6)	0.0433 (6)	−0.0104 (5)	0.0084 (4)	−0.0078 (5)
O2	0.0722 (8)	0.0922 (10)	0.0712 (8)	−0.0402 (7)	0.0275 (6)	−0.0053 (7)
O3	0.0974 (10)	0.0914 (10)	0.0454 (7)	−0.0122 (8)	0.0061 (6)	−0.0174 (7)

Geometric parameters (Å, º)

C2—O3	1.1971 (19)	C9—C10	1.374 (3)
C2—O1	1.3484 (19)	C9—H9	0.9300
C2—C3	1.489 (2)	C10—C11	1.370 (2)
C3—C4	1.506 (2)	C10—H10	0.9300
C3—H3A	0.9700	C11—C12	1.384 (2)
C3—H3B	0.9700	C11—H11	0.9300
C4—O2	1.2013 (19)	C12—H12	0.9300
C4—C5	1.5191 (19)	C13—C18	1.375 (2)
C5—C7	1.5122 (18)	C13—C14	1.382 (2)
C5—C6	1.5353 (19)	C14—C15	1.378 (3)
C5—H5	0.9800	C14—H14	0.9300
C6—O1	1.4633 (16)	C15—C16	1.358 (4)
C6—C13	1.5013 (18)	C15—H15	0.9300
C6—H6	0.9800	C16—C17	1.367 (4)
C7—C8	1.381 (2)	C16—H16	0.9300
C7—C12	1.387 (2)	C17—C18	1.398 (3)
C8—C9	1.382 (2)	C17—H17	0.9300
C8—H8	0.9300	C18—H18	0.9300
O3—C2—O1	119.27 (16)	C10—C9—H9	119.9
O3—C2—C3	124.18 (16)	C8—C9—H9	119.9
O1—C2—C3	116.55 (13)	C11—C10—C9	120.03 (16)
C2—C3—C4	115.28 (13)	C11—C10—H10	120.0
C2—C3—H3A	108.5	C9—C10—H10	120.0
C4—C3—H3A	108.5	C10—C11—C12	119.91 (15)
C2—C3—H3B	108.5	C10—C11—H11	120.0
C4—C3—H3B	108.5	C12—C11—H11	120.0
H3A—C3—H3B	107.5	C11—C12—C7	120.60 (14)
O2—C4—C3	121.58 (14)	C11—C12—H12	119.7
O2—C4—C5	123.07 (15)	C7—C12—H12	119.7
C3—C4—C5	115.35 (12)	C18—C13—C14	119.27 (15)
C7—C5—C4	113.59 (11)	C18—C13—C6	120.03 (14)
C7—C5—C6	112.70 (11)	C14—C13—C6	120.69 (14)
C4—C5—C6	108.47 (11)	C15—C14—C13	120.3 (2)
C7—C5—H5	107.2	C15—C14—H14	119.9
C4—C5—H5	107.2	C13—C14—H14	119.9
C6—C5—H5	107.2	C16—C15—C14	120.5 (2)
O1—C6—C13	106.88 (10)	C16—C15—H15	119.8
O1—C6—C5	109.24 (11)	C14—C15—H15	119.8
C13—C6—C5	113.39 (11)	C15—C16—C17	120.2 (2)

O1—C6—H6	109.1	C15—C16—H16	119.9
C13—C6—H6	109.1	C17—C16—H16	119.9
C5—C6—H6	109.1	C16—C17—C18	119.9 (2)
C8—C7—C12	118.76 (13)	C16—C17—H17	120.0
C8—C7—C5	120.68 (13)	C18—C17—H17	120.0
C12—C7—C5	120.54 (13)	C13—C18—C17	119.8 (2)
C7—C8—C9	120.43 (15)	C13—C18—H18	120.1
C7—C8—H8	119.8	C17—C18—H18	120.1
C9—C8—H8	119.8	C2—O1—C6	118.00 (11)
C10—C9—C8	120.25 (16)		

O3—C2—C3—C4	141.51 (18)	C9—C10—C11—C12	0.3 (3)
O1—C2—C3—C4	−38.6 (2)	C10—C11—C12—C7	0.6 (3)
C2—C3—C4—O2	−153.41 (17)	C8—C7—C12—C11	−1.0 (2)
C2—C3—C4—C5	26.3 (2)	C5—C7—C12—C11	−179.25 (14)
O2—C4—C5—C7	−33.0 (2)	O1—C6—C13—C18	119.45 (14)
C3—C4—C5—C7	147.27 (13)	C5—C6—C13—C18	−120.13 (15)
O2—C4—C5—C6	−159.12 (16)	O1—C6—C13—C14	−61.63 (17)
C3—C4—C5—C6	21.12 (17)	C5—C6—C13—C14	58.80 (18)
C7—C5—C6—O1	173.61 (10)	C18—C13—C14—C15	2.1 (3)
C4—C5—C6—O1	−59.72 (13)	C6—C13—C14—C15	−176.85 (17)
C7—C5—C6—C13	54.54 (15)	C13—C14—C15—C16	−0.9 (3)
C4—C5—C6—C13	−178.80 (11)	C14—C15—C16—C17	−0.5 (4)
C4—C5—C7—C8	126.23 (15)	C15—C16—C17—C18	0.6 (4)
C6—C5—C7—C8	−109.89 (15)	C14—C13—C18—C17	−1.9 (3)
C4—C5—C7—C12	−55.59 (18)	C6—C13—C18—C17	176.99 (16)
C6—C5—C7—C12	68.29 (16)	C16—C17—C18—C13	0.6 (3)
C12—C7—C8—C9	0.5 (2)	O3—C2—O1—C6	177.79 (15)
C5—C7—C8—C9	178.72 (15)	C3—C2—O1—C6	−2.1 (2)
C7—C8—C9—C10	0.4 (3)	C13—C6—O1—C2	175.37 (12)
C8—C9—C10—C11	−0.9 (3)	C5—C6—O1—C2	52.33 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2^{i}	0.98	2.44	3.380 (2)	161
C17—H17···O3^{ii}	0.93	2.46	3.351 (3)	160
C3—H3*B*···*Cg*1^{i}	0.97	2.98	3.681 (2)	131
C5—H5···*Cg*2^{iii}	0.98	2.96	3.830 (2)	149

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