Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Ethoxyphenyl 4-[(methoxycarbonyl)-oxy]benzoate

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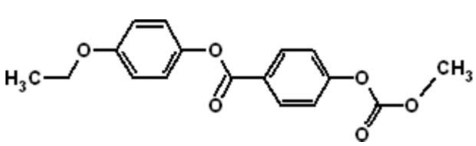
Received 13 August 2010; accepted 1 November 2010

Key indicators: single-crystal X-ray study; *T* = 293 K; mean σ(C–C) = 0.005 Å; *R* factor = 0.035; *wR* factor = 0.085; data-to-parameter ratio = 8.5.

In the title compound, $C_{17}H_{16}O_6$, the two benzene rings form a dihedral angle of 54.95 (10)°. Only weak intermolecular interactions are present in the crystal structure, *viz.* C—H···O hydrogen bonds and C—H···π interactions involving one of the benzene rings.

Related literature

For general background to methoxycarbonyl(oxy)benzoates, see Petrov (2002); Goodby *et al.* (1998); Castellano *et al.* (1971).



Experimental

Crystal data

$C_{17}H_{16}O_6$
M_r = 316.30
Monoclinic, *Cc*
a = 11.7397 (5) Å
b = 16.9703 (6) Å
c = 7.9324 (3) Å
β = 96.949 (4)°
V = 1568.73 (11) Å^3
Z = 4
Mo *Kα* radiation
μ = 0.10 mm^{-1}
T = 293 K
0.22 × 0.15 × 0.12 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Absorption correction: multi-scan (*CrysAlis PRO RED*; Oxford Diffraction, 2009)
T_{min} = 0.982, T_{max} = 0.988
8839 measured reflections
1797 independent reflections
1092 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.085
S = 0.98
1797 reflections
211 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

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

*Cg*1 is the centroid of the benzene ring C2,C4–C8.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16*B*···O2^{i}	0.97	2.56	3.399 (4)	145
C1—H1*B*···*Cg*1^{ii}	0.96	2.99	3.853 (4)	151

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

Data collection: *CrysAlis PRO CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO CCD*; data reduction: *CrysAlis PRO RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank Professor T. N. Guru Row and Mr Venkatesha R. Hathwar, Solid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore, for their help with the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FB2212).

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supporting information

Acta Cryst. (2010). E**66**, o3074 [https://doi.org/10.1107/S1600536810044661]

4-Ethoxyphenyl 4-[(methoxycarbonyl)oxy]benzoate

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S1. Comment

Liquid crystals play important role in technology. Among others, uniaxial calamitic (rod-like) nematic liquid crystals are active switching ingredients for the current LCD technology. A comparative study of physico-chemical and electro-optical properties of achiral calamitic liquid crystals which terminal, bridging and lateral alkoxy groups, with the corresponding alkyl group substituents has been carried out by Petrov (2002). It is well known that the terminal alkoxy substituent does not substantially affect the mesophase behaviour (Goodby *et al.*, 1998). Methyl and propyl group derivatives with respect to the title compound, *i. e.* 4-methoxyphenyl 4-[(methoxycarbonyl)oxy]benzoate and 4-propoxyphenyl 4-[(methoxycarbonyl)oxy]benzoate, respectively, do not form liquid-crystal phase. On the other hand, the title compound with the ethyl group forms a stable nematic phase (Castellano *et al.*, 1971). With this background, we have synthesized the title compound, 4-ethoxyphenyl 4-[(methoxycarbonyl)oxy]benzoate, and herein we report its crystal structure.

The asymmetric unit of the 4-ethoxyphenyl 4-[(methoxycarbonyl)oxy] benzoate, $C_{17}H_{16}O_6$, contains just one molecule (Fig. 1). The two aromatic rings are non-coplanar; the interplanar angle between the two benzene rings (C3\C4\···\C8) and (C10\C11\···C15) equals to 54.95 (10)°. There are only weak intermolecular interactions in the structure: C—H···O hydrogen bonds (Tab. 1) as well as one C—H···*π*-ring electron ring interaction (Tab. 1). The packing of the molecules in the title structure is depicted in Fig. 2.

S2. Experimental

The 4-ethoxyphenyl 4-[(methoxycarbonyl)oxy]benzoate was synthesized according to the procedure described by Castellano *et al.* (1971). The crude white material was subjected to column chromatography using 60–120 mesh silica gel with ethyl acetate (1 ml) and hexane (99 ml) as an eluent. The retention factor equalled to 0.84. Single crystals, suitable for X-ray diffraction analysis, were obtained by recrystallization from pure hexane at room temperature. The yield was about 85%. M.p. 356 K.

Spectral data IR (KBr) cm^{-1}: 2924 and 2852(CH_2 aliphatic), 1759(OC═O ester), 1732(C═O ester), 1604 (aryl C═C), 1462(CH aryl). Elemental analysis: Theor.: C 64.55%, H 5.10%; Found: C 65.01%, H 4.72%.

S3. Refinement

All the H atoms were observable in the difference electron density maps except for just one H from each triplet of the methyl H atoms. Nevertheless, the H atoms were situated into the idealized positions and constrained by the riding atom approximation. The constraints: C_{aryl}—H_{aryl} = 0.93, $C_{methylene}$—$H_{methylene}$ = 0.97 and C_{methyl}—H_{methyl} = 0.96 Å. $U_{iso}(H_{methyl}) = 1.5U_{eq}(C_{methyl})$ or $U_{iso}(H_{methylene/aryl}) = 1.2U_{eq}(C_{methylene/aryl})$. In the absence of significant anomalous scattering effects, 1654 Friedel pairs were merged.

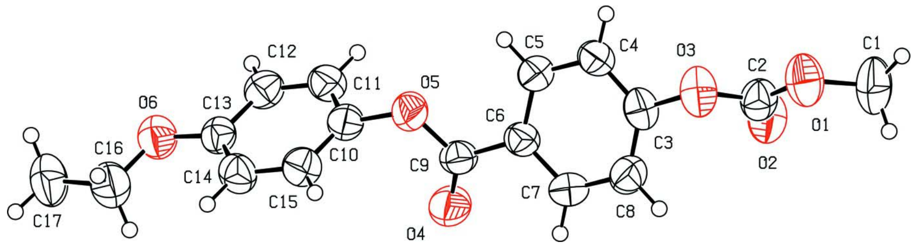

Figure 1

The title molecule with the displacement ellipsoids drawn at the 50% probability level. The H atoms are shown as spheres of arbitrary radii.

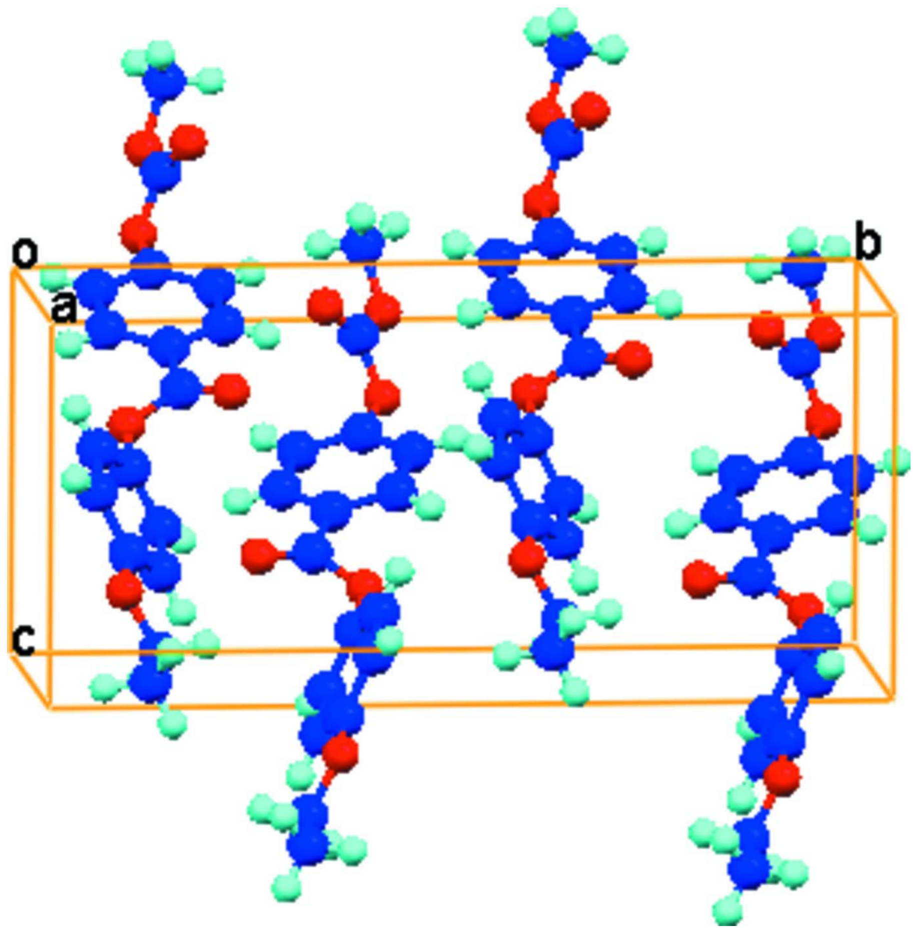

Figure 2

A view of the structure along the axis *a*.

4-Ethoxyphenyl 4-[(methoxycarbonyl)oxy]benzoate

Crystal data

$C_{17}H_{16}O_6$
M_r = 316.30
Monoclinic, *Cc*
Hall symbol: C -2yc
a = 11.7397 (5) Å
b = 16.9703 (6) Å
c = 7.9324 (3) Å
β = 96.949 (4)°
V = 1568.73 (11) Å^3
Z = 4

$F(000) = 664$
$D_x = 1.339$ Mg m^{-3}
Melting point: 356 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1797 reflections
$\theta = 2.4$–$27.5°$
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Plate, colourless
0.22 × 0.15 × 0.12 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0839 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO RED*; Oxford Diffraction, 2009)
$T_{min} = 0.982$, $T_{max} = 0.988$
8839 measured reflections
1797 independent reflections
1092 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
$h = -14 \rightarrow 15$
$k = -21 \rightarrow 21$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.085$
$S = 0.98$
1797 reflections
211 parameters
2 restraints
62 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0418P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.11$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.0052 (7)

Special details

Experimental. *CrysAlisPro*, Oxford Diffraction (2010), Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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	0.75904 (19)	0.40401 (15)	0.0153 (3)	0.0827 (7)
O2	0.5908 (2)	0.34329 (14)	0.0242 (3)	0.0794 (7)
O3	0.68466 (18)	0.40619 (14)	0.2481 (3)	0.0749 (6)
O4	0.3150 (2)	0.27835 (12)	0.7091 (3)	0.0812 (7)
O5	0.3405 (2)	0.40346 (11)	0.7901 (3)	0.0681 (6)
O6	0.03480 (19)	0.39014 (13)	1.2645 (3)	0.0725 (6)
C1	0.7605 (3)	0.3799 (3)	−0.1598 (5)	0.0961 (12)

H1A	0.8087	0.4149	−0.2145	0.144*
H1B	0.6839	0.3815	−0.2180	0.144*
H1C	0.7898	0.3271	−0.1629	0.144*
C2	0.6705 (3)	0.37896 (19)	0.0872 (4)	0.0630 (8)
C3	0.5995 (3)	0.3876 (2)	0.3513 (3)	0.0604 (8)
C4	0.5405 (3)	0.44911 (18)	0.4107 (4)	0.0610 (8)
H4	0.5522	0.5002	0.3743	0.073*
C5	0.4636 (2)	0.43453 (17)	0.5251 (4)	0.0587 (8)
H5	0.4253	0.4763	0.5693	0.070*
C6	0.4428 (2)	0.35787 (17)	0.5747 (3)	0.0523 (7)
C7	0.5019 (3)	0.29643 (17)	0.5101 (4)	0.0610 (8)
H7	0.4877	0.2449	0.5415	0.073*
C8	0.5816 (3)	0.31082 (18)	0.3996 (4)	0.0653 (8)
H8	0.6224	0.2696	0.3583	0.078*
C9	0.3606 (2)	0.34004 (17)	0.6952 (4)	0.0549 (7)
C10	0.2640 (3)	0.39527 (16)	0.9136 (4)	0.0577 (8)
C11	0.1571 (3)	0.42874 (17)	0.8805 (4)	0.0639 (8)
H11	0.1354	0.4534	0.7769	0.077*
C12	0.0830 (3)	0.42581 (17)	0.9999 (4)	0.0620 (8)
H12	0.0105	0.4482	0.9774	0.074*
C13	0.1157 (3)	0.38941 (16)	1.1553 (4)	0.0530 (7)
C14	0.2230 (3)	0.35663 (17)	1.1868 (4)	0.0635 (8)
H14	0.2455	0.3323	1.2905	0.076*
C15	0.2983 (3)	0.35972 (17)	1.0643 (4)	0.0628 (8)
H15	0.3712	0.3377	1.0855	0.075*
C16	0.0587 (3)	0.3503 (2)	1.4190 (5)	0.0816 (10)
H16A	0.1238	0.3744	1.4868	0.098*
H16B	0.0773	0.2957	1.3989	0.098*
C17	−0.0464 (3)	0.3550 (2)	1.5115 (5)	0.0886 (12)
H17A	−0.0310	0.3294	1.6198	0.133*
H17B	−0.1097	0.3294	1.4454	0.133*
H17C	−0.0654	0.4093	1.5281	0.133*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0645 (15)	0.1165 (18)	0.0703 (16)	−0.0045 (12)	0.0212 (12)	−0.0007 (14)
O2	0.0704 (15)	0.1012 (17)	0.0680 (14)	−0.0119 (13)	0.0132 (12)	−0.0162 (13)
O3	0.0674 (15)	0.0997 (16)	0.0590 (14)	−0.0168 (11)	0.0135 (11)	−0.0106 (12)
O4	0.1030 (17)	0.0546 (13)	0.0918 (16)	−0.0184 (11)	0.0355 (13)	−0.0090 (11)
O5	0.0850 (15)	0.0569 (12)	0.0676 (14)	−0.0092 (11)	0.0297 (12)	−0.0046 (11)
O6	0.0739 (15)	0.0778 (14)	0.0674 (15)	−0.0020 (11)	0.0156 (12)	0.0002 (12)
C1	0.085 (3)	0.138 (3)	0.071 (2)	0.009 (2)	0.033 (2)	−0.009 (2)
C2	0.053 (2)	0.075 (2)	0.062 (2)	0.0094 (17)	0.0106 (17)	−0.0008 (18)
C3	0.058 (2)	0.075 (2)	0.0478 (19)	−0.0021 (15)	0.0066 (15)	0.0024 (16)
C4	0.079 (2)	0.0527 (17)	0.0517 (17)	−0.0014 (15)	0.0077 (16)	0.0049 (14)
C5	0.0702 (19)	0.0516 (18)	0.0547 (19)	0.0049 (14)	0.0093 (16)	−0.0020 (14)
C6	0.0586 (18)	0.0494 (18)	0.0479 (18)	0.0016 (13)	0.0025 (15)	0.0032 (13)

C7	0.0687 (19)	0.0477 (17)	0.067 (2)	0.0072 (14)	0.0084 (16)	0.0037 (14)
C8	0.073 (2)	0.064 (2)	0.0593 (18)	0.0156 (15)	0.0096 (16)	−0.0022 (15)
C9	0.0625 (18)	0.0454 (17)	0.0558 (18)	0.0004 (14)	0.0033 (15)	−0.0005 (14)
C10	0.066 (2)	0.0492 (17)	0.061 (2)	−0.0088 (14)	0.0169 (16)	−0.0036 (14)
C11	0.079 (2)	0.0572 (17)	0.0544 (19)	0.0038 (16)	0.0036 (17)	0.0043 (13)
C12	0.0573 (18)	0.0614 (17)	0.067 (2)	0.0067 (14)	0.0057 (16)	−0.0013 (15)
C13	0.0556 (18)	0.0513 (17)	0.0528 (19)	−0.0075 (13)	0.0094 (15)	−0.0069 (14)
C14	0.071 (2)	0.068 (2)	0.0498 (18)	−0.0017 (16)	0.0004 (16)	0.0023 (15)
C15	0.0602 (18)	0.0637 (18)	0.064 (2)	0.0005 (14)	0.0072 (16)	0.0007 (16)
C16	0.093 (3)	0.087 (2)	0.066 (2)	−0.003 (2)	0.016 (2)	0.000 (2)
C17	0.090 (3)	0.110 (3)	0.071 (2)	−0.017 (2)	0.031 (2)	−0.003 (2)

Geometric parameters (Å, º)

O1—C2	1.315 (4)	C6—C9	1.470 (4)
O1—C1	1.450 (4)	C7—C8	1.378 (4)
O2—C2	1.174 (4)	C7—H7	0.9300
O3—C2	1.349 (4)	C8—H8	0.9300
O3—C3	1.403 (3)	C10—C15	1.356 (4)
O4—C9	1.187 (3)	C10—C11	1.373 (4)
O5—C9	1.350 (3)	C11—C12	1.362 (4)
O5—C10	1.414 (3)	C11—H11	0.9300
O6—C13	1.361 (3)	C12—C13	1.390 (4)
O6—C16	1.398 (4)	C12—H12	0.9300
C1—H1A	0.9600	C13—C14	1.373 (4)
C1—H1B	0.9600	C14—C15	1.391 (4)
C1—H1C	0.9600	C14—H14	0.9300
C3—C4	1.368 (4)	C15—H15	0.9300
C3—C8	1.381 (4)	C16—C17	1.512 (5)
C4—C5	1.378 (4)	C16—H16A	0.9700
C4—H4	0.9300	C16—H16B	0.9700
C5—C6	1.389 (4)	C17—H17A	0.9600
C5—H5	0.9300	C17—H17B	0.9600
C6—C7	1.385 (4)	C17—H17C	0.9600
C2—O1—C1	115.2 (3)	O4—C9—C6	125.5 (3)
C2—O3—C3	117.4 (2)	O5—C9—C6	111.8 (2)
C9—O5—C10	118.4 (2)	C15—C10—C11	121.3 (3)
C13—O6—C16	118.2 (3)	C15—C10—O5	120.6 (3)
O1—C1—H1A	109.5	C11—C10—O5	118.0 (3)
O1—C1—H1B	109.5	C12—C11—C10	119.8 (3)
H1A—C1—H1B	109.5	C12—C11—H11	120.1
O1—C1—H1C	109.5	C10—C11—H11	120.1
H1A—C1—H1C	109.5	C11—C12—C13	120.1 (3)
H1B—C1—H1C	109.5	C11—C12—H12	119.9
O2—C2—O1	127.8 (3)	C13—C12—H12	119.9
O2—C2—O3	125.4 (3)	O6—C13—C14	125.7 (3)
O1—C2—O3	106.7 (3)	O6—C13—C12	114.9 (3)

C4—C3—C8	121.6 (3)	C14—C13—C12	119.4 (3)
C4—C3—O3	117.1 (3)	C13—C14—C15	120.3 (3)
C8—C3—O3	121.2 (3)	C13—C14—H14	119.9
C3—C4—C5	119.3 (3)	C15—C14—H14	119.9
C3—C4—H4	120.4	C10—C15—C14	119.1 (3)
C5—C4—H4	120.4	C10—C15—H15	120.5
C4—C5—C6	120.4 (3)	C14—C15—H15	120.5
C4—C5—H5	119.8	O6—C16—C17	108.1 (3)
C6—C5—H5	119.8	O6—C16—H16A	110.1
C7—C6—C5	119.2 (3)	C17—C16—H16A	110.1
C7—C6—C9	118.9 (3)	O6—C16—H16B	110.1
C5—C6—C9	121.9 (2)	C17—C16—H16B	110.1
C8—C7—C6	120.7 (3)	H16A—C16—H16B	108.4
C8—C7—H7	119.6	C16—C17—H17A	109.5
C6—C7—H7	119.6	C16—C17—H17B	109.5
C7—C8—C3	118.8 (3)	H17A—C17—H17B	109.5
C7—C8—H8	120.6	C16—C17—H17C	109.5
C3—C8—H8	120.6	H17A—C17—H17C	109.5
O4—C9—O5	122.7 (3)	H17B—C17—H17C	109.5

C1—O1—C2—O2	−4.3 (5)	C5—C6—C9—O4	158.2 (3)
C1—O1—C2—O3	179.3 (3)	C7—C6—C9—O5	158.5 (3)
C3—O3—C2—O2	2.9 (5)	C5—C6—C9—O5	−20.9 (3)
C3—O3—C2—O1	179.4 (3)	C9—O5—C10—C15	77.9 (3)
C2—O3—C3—C4	−117.8 (3)	C9—O5—C10—C11	−106.2 (3)
C2—O3—C3—C8	66.3 (4)	C15—C10—C11—C12	−0.7 (4)
C8—C3—C4—C5	1.8 (4)	O5—C10—C11—C12	−176.5 (2)
O3—C3—C4—C5	−174.0 (3)	C10—C11—C12—C13	0.4 (4)
C3—C4—C5—C6	−2.4 (4)	C16—O6—C13—C14	−4.1 (4)
C4—C5—C6—C7	1.1 (4)	C16—O6—C13—C12	176.0 (3)
C4—C5—C6—C9	−179.5 (3)	C11—C12—C13—O6	179.9 (2)
C5—C6—C7—C8	0.8 (4)	C11—C12—C13—C14	0.0 (4)
C9—C6—C7—C8	−178.6 (3)	O6—C13—C14—C15	−180.0 (2)
C6—C7—C8—C3	−1.5 (5)	C12—C13—C14—C15	−0.1 (4)
C4—C3—C8—C7	0.1 (4)	C11—C10—C15—C14	0.6 (4)
O3—C3—C8—C7	175.8 (3)	O5—C10—C15—C14	176.3 (2)
C10—O5—C9—O4	1.5 (4)	C13—C14—C15—C10	−0.2 (4)
C10—O5—C9—C6	−179.3 (2)	C13—O6—C16—C17	−176.2 (3)
C7—C6—C9—O4	−22.4 (4)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the benzene ring C2,C4–C8.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16*B*···O2^{i}	0.97	2.56	3.399 (4)	145
C1—H1*B*···*Cg*1^{ii}	0.96	2.99	3.853 (4)	151

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