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4-Bromo-2-(diethoxymethyl)phenyl benzoate

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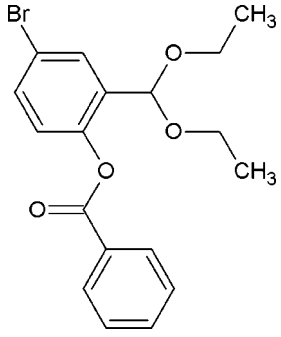
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.085; data-to-parameter ratio = 21.5.

In the title compound, $C_{18}H_{19}BrO_4$, the aromatic rings enclose a dihedral angle of 81.9 (7)°. There are no short directional contacts in the crystal structure.

Related literature

For the biological activity of ester derivatives, see: Bi *et al.* (2012); Bartzatt *et al.* (2004); Anadu *et al.* (2006).



Experimental

Crystal data

$C_{18}H_{19}BrO_4$
M_r = 379.24
Triclinic, $P\overline{1}$
a = 8.2662 (8) Å
b = 9.6378 (10) Å
c = 11.6224 (13) Å
α = 99.927 (5)°
β = 93.700 (5)°
γ = 101.178 (5)°
V = 890.16 (16) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.33 mm^{-1}
T = 293 K
0.35 × 0.30 × 0.25 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker 2004)
T_{min} = 0.497, T_{max} = 0.594
16152 measured reflections
4493 independent reflections
3037 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.085
S = 1.01
4493 reflections
209 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); 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, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6892).

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supplementary materials

Acta Cryst. (2013). E**69**, o553 [doi:10.1107/S1600536813006351]

4-Bromo-2-(diethoxymethyl)phenyl benzoate

P. Sharmila, C. Suresh Kumar, S. Maheshwaran, S. Narasimhan and S. Aravindhan

Comment

Ester derivatives of many compounds exhibit a variety of pharmacological properties, for example anticancer, antitumor and antimicrobial activities (Anadu *et al.*, 2006; Bi *et al.*, 2012; Bartzatt *et al.*, 2004). In view of their importance, the title compound was synthesized and we report herein on its crystal structure.

In the title molecule (Fig. 1) the two aromatic rings enclose a dihedral angle of 98.1 (7)°. The molecular conformation is stabilized by C-H···O contacts. The crystals packing, on the other hand, shows no short contacts.

A packing diagram of the molecule is shown in Fig. 2.

Experimental

A solution of 4-Bromo-2-diethoxymethyl-phenol (0.03 mol) in chloroform (100 ml) was cooled and Benzoyl chlorides (0.03 mol) was added dropwise followed by addition of triethyl amine (0.03 mol). Then, the reaction was stirred at room temperature for 3 h. The reaction mixture was quenched with water and the chloroform layer was separated. The combined chloroform layer was washed with 5% NaOH solution followed by water wash and dried with anhydrous sodium sulfate, concentrated under reduced pressure. The obtained solid was crystallized in a mixture of Methanol:Chloroform.

Refinement

All the H atoms were positioned geometrically, with C–H = 0.93–0.97 Å and constrained to ride on their parent atom, with U_{iso}(H) =1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms.

Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); 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, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009).

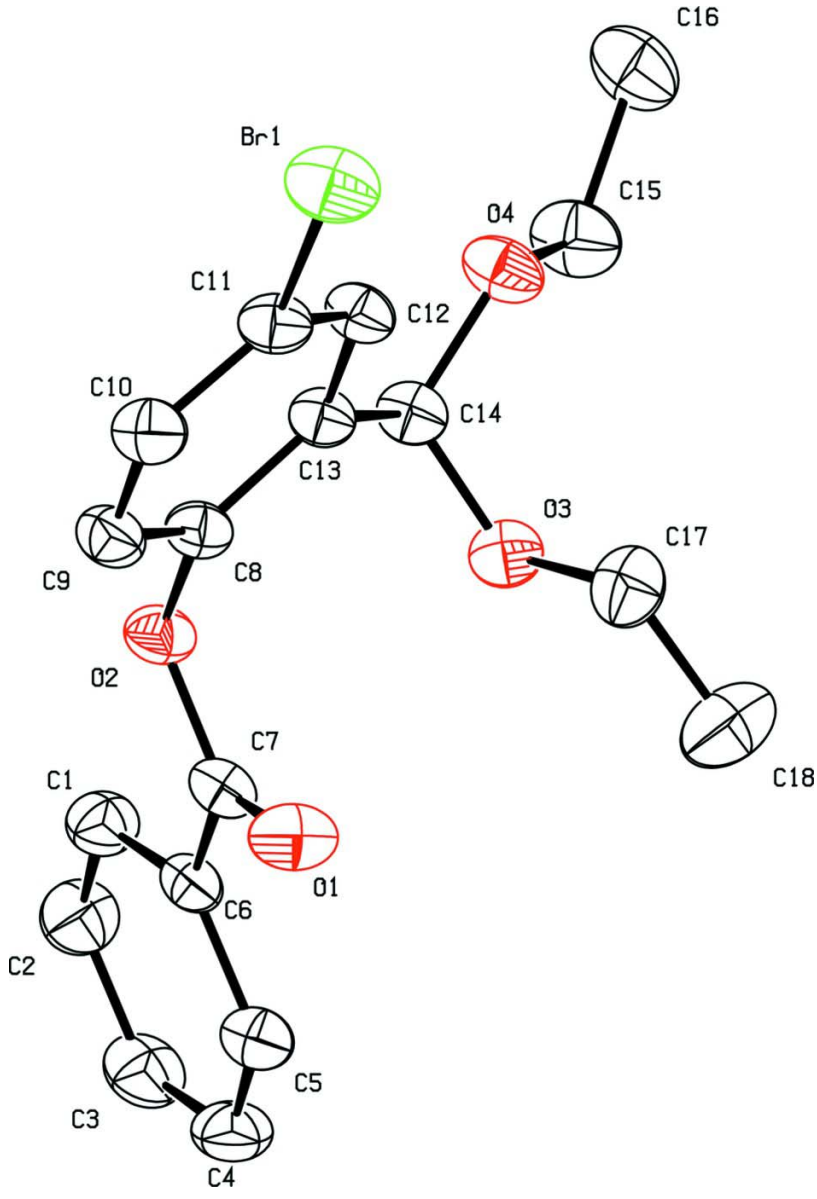

Figure 1

The molecular structure of the title compound, with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

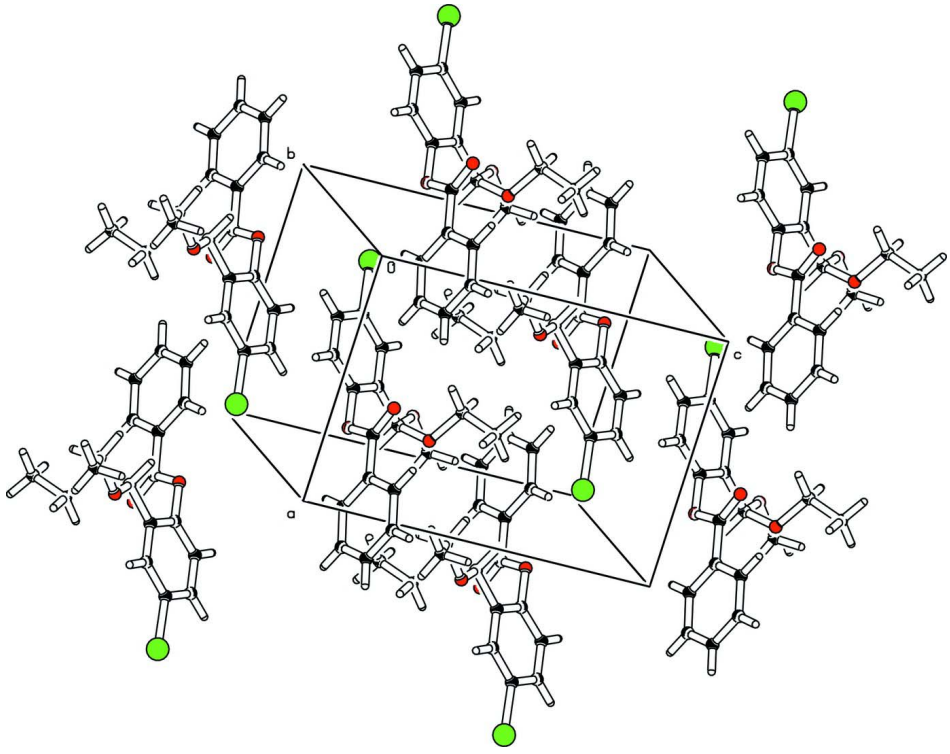

Figure 2

Crystal packing of the title compound viewed along the *b* axis. For the sake of clarity, H atoms have been omitted.

4-Bromo-2-(diethoxymethyl)phenyl benzoate

Crystal data

$C_{18}H_{19}BrO_4$
$M_r = 379.24$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.2662$ (8) Å
$b = 9.6378$ (10) Å
$c = 11.6224$ (13) Å
$\alpha = 99.927$ (5)°
$\beta = 93.700$ (5)°
$\gamma = 101.178$ (5)°
$V = 890.16$ (16) Å^3
$Z = 2$
$F(000) = 388$
$D_x = 1.415$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8834 reflections
$\theta = 2.1$–$31.2°$
$\mu = 2.33$ mm^{-1}
$T = 293$ K
Block, colourless
0.35 × 0.30 × 0.25 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (*SADABS*; Bruker 2004)
$T_{min} = 0.497$, $T_{max} = 0.594$
16152 measured reflections
4493 independent reflections
3037 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 28.6°$, $\theta_{min} = 1.8°$
$h = -10 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.085$
$S = 1.01$
4493 reflections
209 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
$w = 1/[\sigma^2(F_o^2) + (0.0325P)^2 + 0.2669P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0293 (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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.0177 (3)	0.1162 (2)	0.14518 (19)	0.0568 (5)
H1	1.0276	0.2024	0.1179	0.068*
C2	1.1551 (3)	0.0569 (3)	0.1588 (2)	0.0668 (6)
H2	1.2575	0.1035	0.1415	0.080*
C3	1.1409 (3)	−0.0706 (3)	0.1979 (2)	0.0686 (6)
H3	1.2335	−0.1108	0.2067	0.082*
C4	0.9906 (3)	−0.1388 (2)	0.2238 (2)	0.0657 (6)
H4	0.9814	−0.2257	0.2500	0.079*
C5	0.8522 (3)	−0.0800 (2)	0.21151 (19)	0.0560 (5)
H5	0.7504	−0.1266	0.2299	0.067*
C6	0.8660 (2)	0.0487 (2)	0.17159 (16)	0.0446 (4)
C7	0.7144 (3)	0.1076 (2)	0.16072 (17)	0.0476 (5)
C8	0.6138 (2)	0.3097 (2)	0.12700 (17)	0.0454 (4)
C9	0.4947 (3)	0.2747 (2)	0.03277 (18)	0.0530 (5)
H9	0.5006	0.2030	−0.0306	0.064*
C10	0.3663 (3)	0.3469 (2)	0.03332 (19)	0.0553 (5)
H10	0.2848	0.3249	−0.0298	0.066*
C11	0.3603 (2)	0.4517 (2)	0.12799 (18)	0.0495 (5)
C12	0.4820 (2)	0.4902 (2)	0.22060 (17)	0.0477 (5)
H12	0.4769	0.5639	0.2827	0.057*
C13	0.6130 (2)	0.4189 (2)	0.22129 (17)	0.0440 (4)
C14	0.7505 (2)	0.4552 (2)	0.32109 (18)	0.0487 (5)
H14	0.8563	0.4704	0.2868	0.058*
C15	0.8756 (3)	0.6431 (3)	0.4779 (2)	0.0728 (7)

H15A	0.9786	0.6372	0.4438	0.087*
H15B	0.8675	0.5876	0.5402	0.087*
C16	0.8736 (4)	0.7952 (3)	0.5262 (2)	0.0831 (8)
H16A	0.9657	0.8344	0.5850	0.125*
H16B	0.7720	0.8003	0.5607	0.125*
H16C	0.8819	0.8497	0.4642	0.125*
C17	0.5999 (3)	0.3033 (3)	0.4376 (2)	0.0698 (6)
H17A	0.5091	0.2600	0.3771	0.084*
H17B	0.5723	0.3875	0.4843	0.084*
C18	0.6251 (4)	0.1991 (4)	0.5128 (3)	0.1005 (10)
H18A	0.5260	0.1717	0.5494	0.151*
H18B	0.7155	0.2424	0.5721	0.151*
H18C	0.6501	0.1153	0.4657	0.151*
O1	0.57904 (18)	0.05021 (16)	0.17780 (15)	0.0657 (4)
O2	0.74639 (16)	0.23833 (14)	0.12732 (12)	0.0502 (3)
O3	0.74890 (16)	0.34407 (15)	0.38518 (12)	0.0535 (3)
O4	0.73826 (17)	0.58602 (15)	0.38984 (13)	0.0578 (4)
Br1	0.17952 (3)	0.54626 (3)	0.13035 (2)	0.07463 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0560 (12)	0.0532 (12)	0.0665 (14)	0.0203 (10)	0.0098 (10)	0.0140 (10)
C2	0.0549 (13)	0.0715 (16)	0.0812 (16)	0.0235 (12)	0.0154 (12)	0.0195 (13)
C3	0.0660 (15)	0.0711 (16)	0.0759 (16)	0.0377 (13)	0.0041 (12)	0.0085 (13)
C4	0.0794 (17)	0.0487 (12)	0.0752 (15)	0.0271 (12)	0.0037 (12)	0.0142 (11)
C5	0.0601 (13)	0.0463 (12)	0.0606 (13)	0.0147 (10)	0.0032 (10)	0.0044 (10)
C6	0.0501 (11)	0.0403 (10)	0.0418 (10)	0.0145 (8)	0.0012 (8)	−0.0010 (8)
C7	0.0491 (11)	0.0401 (10)	0.0499 (11)	0.0116 (9)	0.0018 (9)	−0.0028 (9)
C8	0.0437 (10)	0.0399 (10)	0.0541 (12)	0.0137 (8)	0.0050 (8)	0.0073 (9)
C9	0.0616 (13)	0.0442 (11)	0.0495 (12)	0.0127 (10)	−0.0018 (10)	−0.0005 (9)
C10	0.0558 (12)	0.0502 (12)	0.0557 (12)	0.0102 (10)	−0.0116 (10)	0.0062 (10)
C11	0.0440 (10)	0.0445 (11)	0.0608 (13)	0.0146 (8)	−0.0032 (9)	0.0096 (9)
C12	0.0469 (10)	0.0429 (10)	0.0517 (11)	0.0150 (8)	−0.0022 (9)	0.0007 (9)
C13	0.0402 (9)	0.0417 (10)	0.0495 (11)	0.0105 (8)	−0.0001 (8)	0.0058 (8)
C14	0.0423 (10)	0.0471 (11)	0.0551 (12)	0.0118 (8)	−0.0024 (9)	0.0052 (9)
C15	0.0678 (15)	0.0647 (15)	0.0731 (16)	0.0076 (12)	−0.0277 (12)	−0.0026 (12)
C16	0.0846 (18)	0.0742 (17)	0.0724 (17)	0.0070 (14)	−0.0140 (14)	−0.0166 (13)
C17	0.0651 (15)	0.0801 (17)	0.0676 (15)	0.0161 (13)	0.0103 (12)	0.0209 (13)
C18	0.104 (2)	0.092 (2)	0.116 (2)	0.0156 (18)	0.0205 (19)	0.0523 (19)
O1	0.0468 (8)	0.0520 (9)	0.0981 (12)	0.0104 (7)	0.0066 (8)	0.0144 (8)
O2	0.0469 (7)	0.0433 (7)	0.0639 (9)	0.0181 (6)	0.0090 (6)	0.0086 (6)
O3	0.0459 (7)	0.0551 (8)	0.0607 (8)	0.0136 (6)	−0.0023 (6)	0.0135 (7)
O4	0.0532 (8)	0.0498 (8)	0.0622 (9)	0.0137 (6)	−0.0156 (7)	−0.0073 (7)
Br1	0.05575 (16)	0.07410 (19)	0.0983 (2)	0.03299 (12)	−0.00794 (12)	0.01173 (14)

Geometric parameters (Å, º)

C1—C6	1.375 (3)	C11—Br1	1.8944 (19)
C1—C2	1.380 (3)	C12—C13	1.391 (3)

C1—H1	0.9300	C12—H12	0.9300
C2—C3	1.368 (3)	C13—C14	1.515 (3)
C2—H2	0.9300	C14—O4	1.397 (2)
C3—C4	1.366 (3)	C14—O3	1.405 (2)
C3—H3	0.9300	C14—H14	0.9800
C4—C5	1.382 (3)	C15—O4	1.432 (2)
C4—H4	0.9300	C15—C16	1.480 (3)
C5—C6	1.385 (3)	C15—H15A	0.9700
C5—H5	0.9300	C15—H15B	0.9700
C6—C7	1.480 (3)	C16—H16A	0.9600
C7—O1	1.193 (2)	C16—H16B	0.9600
C7—O2	1.364 (2)	C16—H16C	0.9600
C8—C9	1.374 (3)	C17—O3	1.427 (3)
C8—C13	1.384 (3)	C17—C18	1.475 (4)
C8—O2	1.402 (2)	C17—H17A	0.9700
C9—C10	1.377 (3)	C17—H17B	0.9700
C9—H9	0.9300	C18—H18A	0.9600
C10—C11	1.370 (3)	C18—H18B	0.9600
C10—H10	0.9300	C18—H18C	0.9600
C11—C12	1.374 (3)		

C6—C1—C2	120.4 (2)	C8—C13—C12	117.63 (17)
C6—C1—H1	119.8	C8—C13—C14	119.86 (16)
C2—C1—H1	119.8	C12—C13—C14	122.51 (17)
C3—C2—C1	120.1 (2)	O4—C14—O3	113.44 (17)
C3—C2—H2	120.0	O4—C14—C13	107.35 (15)
C1—C2—H2	120.0	O3—C14—C13	112.85 (15)
C4—C3—C2	120.0 (2)	O4—C14—H14	107.6
C4—C3—H3	120.0	O3—C14—H14	107.6
C2—C3—H3	120.0	C13—C14—H14	107.6
C3—C4—C5	120.6 (2)	O4—C15—C16	109.0 (2)
C3—C4—H4	119.7	O4—C15—H15A	109.9
C5—C4—H4	119.7	C16—C15—H15A	109.9
C4—C5—C6	119.5 (2)	O4—C15—H15B	109.9
C4—C5—H5	120.2	C16—C15—H15B	109.9
C6—C5—H5	120.2	H15A—C15—H15B	108.3
C1—C6—C5	119.40 (18)	C15—C16—H16A	109.5
C1—C6—C7	123.17 (18)	C15—C16—H16B	109.5
C5—C6—C7	117.43 (19)	H16A—C16—H16B	109.5
O1—C7—O2	122.72 (18)	C15—C16—H16C	109.5
O1—C7—C6	125.59 (19)	H16A—C16—H16C	109.5
O2—C7—C6	111.68 (17)	H16B—C16—H16C	109.5
C9—C8—C13	122.30 (17)	O3—C17—C18	108.7 (2)
C9—C8—O2	120.00 (17)	O3—C17—H17A	110.0
C13—C8—O2	117.64 (16)	C18—C17—H17A	110.0
C8—C9—C10	119.24 (18)	O3—C17—H17B	110.0
C8—C9—H9	120.4	C18—C17—H17B	110.0
C10—C9—H9	120.4	H17A—C17—H17B	108.3
C11—C10—C9	119.18 (18)	C17—C18—H18A	109.5

C11—C10—H10	120.4	C17—C18—H18B	109.5
C9—C10—H10	120.4	H18A—C18—H18B	109.5
C10—C11—C12	121.75 (18)	C17—C18—H18C	109.5
C10—C11—Br1	118.92 (14)	H18A—C18—H18C	109.5
C12—C11—Br1	119.33 (15)	H18B—C18—H18C	109.5
C11—C12—C13	119.83 (18)	C7—O2—C8	115.71 (15)
C11—C12—H12	120.1	C14—O3—C17	115.33 (16)
C13—C12—H12	120.1	C14—O4—C15	112.95 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O2	0.93	2.42	2.740 (3)	100
C12—H12···O4	0.93	2.38	2.699 (2)	100
C17—H17*B*···O4	0.97	2.58	2.905 (3)	100