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Diethyl 9,10-*endo*-ethano-9,10-dihydroanthracene-11,11-dicarboxylate

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Key indicators

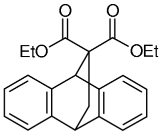
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.055
wR factor = 0.143
Data-to-parameter ratio = 14.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

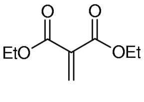
The title compound, $C_{22}H_{22}O_4$, possesses normal geometrical parameters and the dihedral angle between the two benzene ring planes is 57.62 (5)°. The crystal packing is controlled by van der Waals forces and a possible C—H$\cdots$O interaction, the latter resulting in a supramolecular C(6) motif.

Comment

The title compound, (I) (Fig. 1), was created as an intermediate in the synthesis of 2-methylene malonic acid diethyl ester, (II). The alkene produced in the absence of anthracene is very unstable and polymerizes easily. The presence of the anthracene acts to trap the monomer in a Diels–Alder reaction and purification of (I) prior to thermolysis allows the generation of (II) (by a retro-Diels–Alder reaction) in a much more stable form. The presence of excess maleic anhydride in this reaction ensures that the released anthracene is consumed by the formation of an anthracene-maleic anhydride adduct and is not free to regenerate (I). Thus, this type of reaction may be useful in the trapping of alkenes and allow for easier purification.



(I)



(II)

The geometrical parameters for (I) are broadly similar to those of related 9,10-bridged anthracene derivatives (Table 1) (Gable *et al.*, 1996; Karolak-Wojciechowska *et al.*, 1998; Burrows *et al.*, 1999). The two benzene rings in (I) (atoms C2–C7 and C9–C14) are both essentially planar (r.m.s. deviations from the least-squares planes are 0.010 and 0.001 Å, respectively). The dihedral angle between these rings is 57.62 (5)°, which is typical for these 9,10-bridged anthracene systems, *e.g.* the corresponding dihedral angle in 11,12-bis(*N*,*N*-dimethylaminomethyl)-9,10-dihydro-9,10-ethanoanthracene (Karolak-Wojciechowska *et al.*, 1998) is 58.8 (2)°. The three six-membered rings of the bicyclic core of (I) (C1/C2/C7/C8/C9/C14, C1/C2/C7/C8/C15/C16 and C1/C14/C9/C8/C15/C16; see Fig. 1) are all forced into boat forms. The ester substituents show no unusual features.

The only significant intermolecular interaction in (I), as identified in a *PLATON* (Spek, 2003) analysis of the structure, is a C8—H8$\cdots$O3^{i} bond (Table 2). This bridgehead H8 atom attached to an sp^3-hybridized C atom may be slightly activated due to ring strain (Desiraju & Steiner, 1999). This connectivity results in C(6) chains (Bernstein *et al.*, 1995), generated by *n*-

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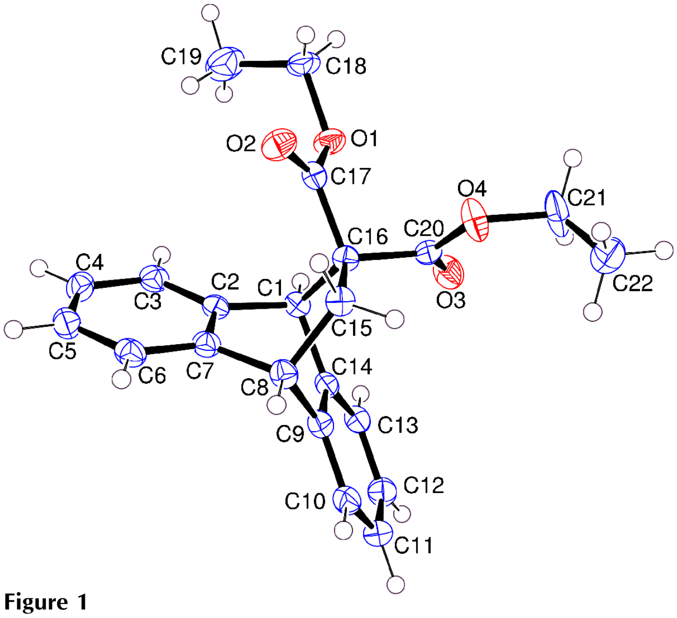

Figure 1
View of (I) (50% displacement ellipsoids). H atoms are drawn as small spheres of arbitrary radius.

glide symmetry (Fig. 2). Otherwise, the crystal packing is controlled by van der Waals forces.

Experimental

A round-bottomed flask was fitted with a still head and condenser and diethyl malonate (9.70 g, 9.2 ml, 61 mmol), anthracene (12.00 g, 67 mmol), paraformaldehyde (3.64 g, 0.12 mol), copper(II) acetate monohydrate (0.60 g, 3.0 mmol), acetic acid (50 ml) and xylene (50 ml) were quickly added. The reaction mixture was heated at 383 K for 15 h and a clear dark-green solution resulted. The temperature was increased in order to distil off the acetic acid, then the reaction mixture was cooled to room temperature and filtered under suction. The filtrate was retained and the xylene evaporated on a rotary evaporator to yield a green oil which was left to crystallize. Purification was carried out by recrystallization from hot hexane. Filtration and washing with ice-cold hexane (25 ml) resulted in the pure anthracene adduct (I) (14.72 g, 69%) as colourless plates [m.p. 404.5–405 K; literature (De Keyser *et al.*, 1988) 403–404 K from EtOH]; R_F(hexane–propan-2-ol 50:1) 0.13; ν_{max} (KBr disc)/cm^{-1}: 2974 (C—H), 1732 (C═O), 1460–1446 (aromatic C═C) and 757 (4 adjacent Ar-H); δH (250 MHz; $CDCl_3$): 1.15 (6H, *t*, J = 7.0 Hz, 2 × CH_3), 2.47 [2H, *d*, J = 2.4 Hz, $(EtO_2C)_2CCH_2$], 3.95–4.09 (4H, *m*, 2 × OCH_2), 4.33 (1H, poorly resolved *t*, J = 2.4 Hz, Ar_2CHCH_2), 4.97 [1H, *s*, $Ar_2CHC(CO_2Et)_2$] and 7.07–7.33 (8H, *m*, Ar-H); δC ($CDCl_3$): 14.0, 36.4, 43.9, 49.6, 60.0, 61.7, 123.3, 125.7, [De Keyser *et al.* (1988) give 125.68 and 125.74], 126.4, 139.8, 144.0 and 170.2.

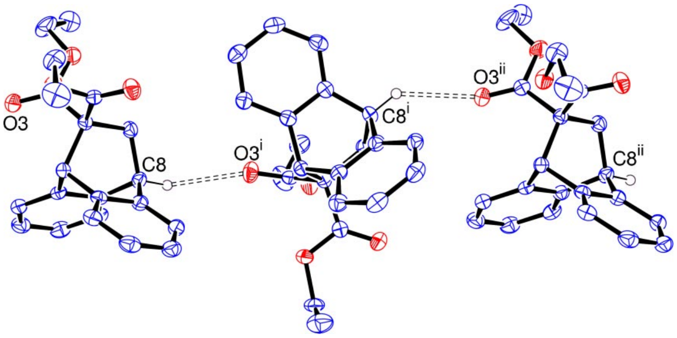

Figure 2
Detail of a chain of molecules of (I) linked by C—H···O interactions. [Symmetry codes: (i) as in Table 2; (ii) $x + 1$, y, $z + 1$.]

Crystal data

$C_{22}H_{22}O_4$
M_r = 350.40
Monoclinic, $P2_1/n$
a = 9.2424 (2) Å
b = 16.5210 (5) Å
c = 11.9154 (4) Å
β = 98.631 (2)°
V = 1798.80 (9) Å^3
Z = 4
D_x = 1.294 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 21 198 reflections
θ = 2.9–27.5°
μ = 0.09 mm^{-1}
T = 120 (2) K
Plate, colourless
0.28 × 0.20 × 0.03 mm

Data collection

Enraf–Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.976, T_{max} = 0.999
21 237 measured reflections
3538 independent reflections
2750 reflections with $I > 2\sigma(I)$
R_{int} = 0.120
θ_{max} = 26.0°
$h = -11 \rightarrow 11$
$k = -20 \rightarrow 20$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.143
S = 1.06
3538 reflections
238 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0607P)^2 + 0.8446P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.012 (3)

Table 1
Selected geometrical parameters (Å °).

C1—C16	1.576 (3)	C15—C16	1.559 (3)
C8—C15	1.550 (3)		
C20—C16—C17—O2	−131.2 (2)	C17—C16—C20—O3	−109.9 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O3^i	1.00	2.56	3.380 (2)	139

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

All H atoms were geometrically placed in idealized locations and refined as riding on their carrier C atoms with C—H distances set to 0.95, 0.98, 0.99 and 1.00 Å for aromatic, sp^2, terminal sp^3 and bridgehead sp^3 hybrid C atoms, respectively. The constraint U_{iso}(H) = 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(methyl C) was applied as appropriate.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *COLLECT* and *DENZO* (Otwinowski & Minor, 1997); data reduction: *COLLECT* and *DENZO*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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supporting information

Acta Cryst. (2004). E**60**, o1081–o1083   [https://doi.org/10.1107/S1600536804012279]

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(I)

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$D_x = 1.294$ Mg m^{-3}
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Cell parameters from 21198 reflections
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Radiation source: fine-focus sealed tube
Graphite monochromator
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Absorption correction: multi-scan (SORTAV; Blessing, 1995)
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21237 measured reflections
3538 independent reflections
2750 reflections with $I > 2\sigma(I)'$
$R_{\rm int} = 0.120$
$\theta_{\max} = 26.0°$, $\theta_{\min} = 3.0°$
$h = -11 \rightarrow 11$
$k = -20 \rightarrow 20$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.143$
$S = 1.06$
3538 reflections
238 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
$w = 1/[\sigma^2(F_o^2) + (0.0607P)^2 + 0.8446P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.012 (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	$U_{\rm iso}$*/$U_{\rm eq}$
C1	0.0216 (2)	0.24941 (11)	0.79618 (16)	0.0207 (4)
H1	0.0063	0.2266	0.7176	0.025*
C2	0.1800 (2)	0.27254 (11)	0.83512 (16)	0.0221 (4)
C3	0.2947 (2)	0.26234 (12)	0.77362 (17)	0.0267 (5)
H3	0.2785	0.2375	0.7009	0.032*
C4	0.4342 (2)	0.28899 (13)	0.81988 (18)	0.0303 (5)
H4	0.5134	0.2828	0.7782	0.036*
C5	0.4574 (2)	0.32439 (13)	0.92616 (18)	0.0306 (5)
H5	0.5526	0.3426	0.9568	0.037*
C6	0.3427 (2)	0.33370 (12)	0.98894 (17)	0.0272 (5)
H6	0.3596	0.3572	1.0625	0.033*
C7	0.2032 (2)	0.30808 (12)	0.94241 (16)	0.0237 (4)
C8	0.0657 (2)	0.31482 (12)	0.99611 (16)	0.0234 (4)
H8	0.0844	0.3424	1.0716	0.028*
C9	−0.0462 (2)	0.35975 (12)	0.91330 (16)	0.0233 (4)
C10	−0.1241 (2)	0.42843 (12)	0.93459 (18)	0.0279 (5)
H10	−0.1088	0.4528	1.0076	0.033*
C11	−0.2248 (2)	0.46132 (13)	0.84820 (19)	0.0314 (5)
H11	−0.2782	0.5084	0.8622	0.038*
C12	−0.2475 (2)	0.42579 (13)	0.74195 (18)	0.0305 (5)
H12	−0.3166	0.4487	0.6835	0.037*
C13	−0.1706 (2)	0.35706 (12)	0.71985 (17)	0.0247 (4)
H13	−0.1866	0.3328	0.6467	0.030*
C14	−0.0703 (2)	0.32413 (11)	0.80559 (16)	0.0217 (4)
C15	0.0035 (2)	0.22833 (12)	1.00520 (16)	0.0238 (4)
H15A	−0.0908	0.2312	1.0349	0.029*
H15B	0.0725	0.1958	1.0587	0.029*
C16	−0.0199 (2)	0.18699 (11)	0.88621 (15)	0.0210 (4)
C17	0.0766 (2)	0.11174 (12)	0.88462 (15)	0.0220 (4)
C18	0.1230 (2)	−0.00151 (13)	0.77158 (19)	0.0327 (5)
H18A	0.0615	−0.0390	0.7201	0.039*
H18B	0.1501	−0.0290	0.8456	0.039*
C19	0.2581 (3)	0.01865 (16)	0.7228 (2)	0.0473 (6)
H19A	0.3088	−0.0314	0.7079	0.071*

H19B	0.3227	0.0521	0.7767	0.071*
H19C	0.2314	0.0485	0.6516	0.071*
C20	−0.1775 (2)	0.15905 (12)	0.85190 (16)	0.0219 (4)
C21	−0.3694 (2)	0.08520 (17)	0.9188 (2)	0.0441 (6)
H21A	−0.3710	0.0253	0.9166	0.053*
H21B	−0.4223	0.1056	0.8458	0.053*
C22	−0.4421 (3)	0.11405 (15)	1.0129 (2)	0.0402 (6)
H22A	−0.5432	0.0944	1.0026	0.060*
H22B	−0.4419	0.1734	1.0139	0.060*
H22C	−0.3895	0.0937	1.0849	0.060*
O1	0.03986 (15)	0.07194 (8)	0.78719 (12)	0.0287 (4)
O2	0.17203 (16)	0.09130 (9)	0.95894 (12)	0.0321 (4)
O3	−0.25370 (15)	0.17464 (9)	0.76424 (12)	0.0333 (4)
O4	−0.21870 (16)	0.11370 (10)	0.93410 (12)	0.0366 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0227 (9)	0.0208 (10)	0.0182 (9)	−0.0013 (8)	0.0025 (7)	−0.0005 (7)
C2	0.0247 (10)	0.0175 (10)	0.0242 (10)	−0.0001 (8)	0.0043 (8)	0.0042 (8)
C3	0.0307 (11)	0.0228 (11)	0.0270 (11)	0.0004 (8)	0.0062 (8)	0.0043 (8)
C4	0.0245 (10)	0.0291 (12)	0.0387 (12)	0.0025 (9)	0.0092 (9)	0.0099 (9)
C5	0.0236 (10)	0.0271 (12)	0.0396 (12)	−0.0024 (8)	0.0000 (9)	0.0078 (9)
C6	0.0293 (11)	0.0217 (10)	0.0284 (11)	−0.0013 (8)	−0.0032 (8)	0.0022 (8)
C7	0.0245 (10)	0.0199 (10)	0.0258 (10)	0.0004 (8)	0.0014 (8)	0.0019 (8)
C8	0.0244 (10)	0.0248 (10)	0.0205 (10)	−0.0030 (8)	0.0020 (8)	−0.0038 (8)
C9	0.0230 (10)	0.0233 (10)	0.0239 (10)	−0.0035 (8)	0.0047 (7)	−0.0001 (8)
C10	0.0272 (11)	0.0248 (11)	0.0325 (11)	−0.0048 (8)	0.0068 (8)	−0.0069 (9)
C11	0.0277 (11)	0.0226 (11)	0.0446 (13)	0.0022 (9)	0.0078 (9)	−0.0013 (9)
C12	0.0275 (11)	0.0268 (12)	0.0368 (12)	0.0011 (9)	0.0034 (9)	0.0064 (9)
C13	0.0244 (10)	0.0236 (11)	0.0257 (10)	−0.0036 (8)	0.0020 (8)	0.0014 (8)
C14	0.0214 (10)	0.0193 (10)	0.0248 (10)	−0.0044 (8)	0.0049 (8)	−0.0007 (8)
C15	0.0257 (10)	0.0266 (11)	0.0193 (10)	−0.0003 (8)	0.0036 (8)	0.0001 (8)
C16	0.0221 (10)	0.0209 (10)	0.0196 (9)	−0.0001 (7)	0.0022 (7)	−0.0006 (7)
C17	0.0213 (9)	0.0221 (10)	0.0229 (10)	−0.0035 (8)	0.0044 (8)	0.0012 (8)
C18	0.0334 (12)	0.0214 (11)	0.0428 (13)	0.0066 (9)	0.0039 (10)	−0.0087 (9)
C19	0.0495 (15)	0.0438 (15)	0.0524 (15)	0.0110 (12)	0.0203 (12)	0.0001 (12)
C20	0.0232 (10)	0.0199 (10)	0.0232 (10)	0.0018 (8)	0.0053 (8)	−0.0031 (8)
C21	0.0316 (13)	0.0571 (16)	0.0457 (14)	−0.0259 (11)	0.0128 (10)	−0.0100 (12)
C22	0.0332 (12)	0.0418 (14)	0.0464 (14)	0.0044 (10)	0.0083 (10)	0.0025 (11)
O1	0.0286 (8)	0.0245 (8)	0.0319 (8)	0.0049 (6)	0.0005 (6)	−0.0079 (6)
O2	0.0312 (8)	0.0340 (9)	0.0294 (8)	0.0072 (6)	−0.0010 (6)	0.0024 (6)
O3	0.0283 (8)	0.0345 (9)	0.0339 (8)	−0.0054 (6)	−0.0065 (6)	0.0042 (6)
O4	0.0305 (8)	0.0487 (10)	0.0306 (8)	−0.0174 (7)	0.0043 (6)	0.0057 (7)

Geometric parameters (Å, º)

C1—C14	1.512 (3)	C13—C14	1.384 (3)
C1—C2	1.516 (3)	C13—H13	0.9500
C1—C16	1.576 (3)	C15—C16	1.559 (3)
C1—H1	1.0000	C15—H15A	0.9900
C2—C3	1.387 (3)	C15—H15B	0.9900
C2—C7	1.394 (3)	C16—C20	1.524 (3)
C3—C4	1.395 (3)	C16—C17	1.532 (3)
C3—H3	0.9500	C17—O2	1.201 (2)
C4—C5	1.382 (3)	C17—O1	1.333 (2)
C4—H4	0.9500	C18—O1	1.463 (2)
C5—C6	1.395 (3)	C18—C19	1.492 (3)
C5—H5	0.9500	C18—H18A	0.9900
C6—C7	1.391 (3)	C18—H18B	0.9900
C6—H6	0.9500	C19—H19A	0.9800
C7—C8	1.510 (3)	C19—H19B	0.9800
C8—C9	1.513 (3)	C19—H19C	0.9800
C8—C15	1.550 (3)	C20—O3	1.197 (2)
C8—H8	1.0000	C20—O4	1.333 (2)
C9—C10	1.387 (3)	C21—O4	1.455 (2)
C9—C14	1.399 (3)	C21—C22	1.470 (3)
C10—C11	1.390 (3)	C21—H21A	0.9900
C10—H10	0.9500	C21—H21B	0.9900
C11—C12	1.383 (3)	C22—H22A	0.9800
C11—H11	0.9500	C22—H22B	0.9800
C12—C13	1.386 (3)	C22—H22C	0.9800
C12—H12	0.9500		
C14—C1—C2	107.37 (15)	C13—C14—C1	126.00 (17)
C14—C1—C16	106.66 (15)	C9—C14—C1	113.45 (16)
C2—C1—C16	106.21 (14)	C8—C15—C16	110.04 (15)
C14—C1—H1	112.1	C8—C15—H15A	109.7
C2—C1—H1	112.1	C16—C15—H15A	109.7
C16—C1—H1	112.1	C8—C15—H15B	109.7
C3—C2—C7	120.74 (18)	C16—C15—H15B	109.7
C3—C2—C1	125.96 (18)	H15A—C15—H15B	108.2
C7—C2—C1	113.29 (16)	C20—C16—C17	106.61 (15)
C2—C3—C4	119.17 (19)	C20—C16—C15	111.94 (15)
C2—C3—H3	120.4	C17—C16—C15	111.39 (15)
C4—C3—H3	120.4	C20—C16—C1	109.64 (15)
C5—C4—C3	120.18 (19)	C17—C16—C1	108.70 (15)
C5—C4—H4	119.9	C15—C16—C1	108.50 (15)
C3—C4—H4	119.9	O2—C17—O1	124.51 (18)
C4—C5—C6	120.81 (19)	O2—C17—C16	125.72 (17)
C4—C5—H5	119.6	O1—C17—C16	109.77 (15)
C6—C5—H5	119.6	O1—C18—C19	110.52 (18)
C7—C6—C5	119.10 (19)	O1—C18—H18A	109.5

C7—C6—H6	120.4	C19—C18—H18A	109.5
C5—C6—H6	120.4	O1—C18—H18B	109.5
C6—C7—C2	119.98 (18)	C19—C18—H18B	109.5
C6—C7—C8	126.67 (18)	H18A—C18—H18B	108.1
C2—C7—C8	113.34 (16)	C18—C19—H19A	109.5
C7—C8—C9	107.23 (15)	C18—C19—H19B	109.5
C7—C8—C15	107.81 (16)	H19A—C19—H19B	109.5
C9—C8—C15	105.88 (15)	C18—C19—H19C	109.5
C7—C8—H8	111.9	H19A—C19—H19C	109.5
C9—C8—H8	111.9	H19B—C19—H19C	109.5
C15—C8—H8	111.9	O3—C20—O4	124.48 (18)
C10—C9—C14	119.77 (18)	O3—C20—C16	125.63 (18)
C10—C9—C8	127.21 (18)	O4—C20—C16	109.88 (16)
C14—C9—C8	113.00 (17)	O4—C21—C22	109.74 (19)
C9—C10—C11	119.48 (19)	O4—C21—H21A	109.7
C9—C10—H10	120.3	C22—C21—H21A	109.7
C11—C10—H10	120.3	O4—C21—H21B	109.7
C12—C11—C10	120.33 (19)	C22—C21—H21B	109.7
C12—C11—H11	119.8	H21A—C21—H21B	108.2
C10—C11—H11	119.8	C21—C22—H22A	109.5
C11—C12—C13	120.67 (19)	C21—C22—H22B	109.5
C11—C12—H12	119.7	H22A—C22—H22B	109.5
C13—C12—H12	119.7	C21—C22—H22C	109.5
C14—C13—C12	119.20 (18)	H22A—C22—H22C	109.5
C14—C13—H13	120.4	H22B—C22—H22C	109.5
C12—C13—H13	120.4	C17—O1—C18	116.73 (15)
C13—C14—C9	120.55 (18)	C20—O4—C21	117.59 (17)

C14—C1—C2—C3	−125.2 (2)	C2—C1—C14—C13	126.6 (2)
C16—C1—C2—C3	121.0 (2)	C16—C1—C14—C13	−119.9 (2)
C14—C1—C2—C7	53.7 (2)	C2—C1—C14—C9	−53.7 (2)
C16—C1—C2—C7	−60.1 (2)	C16—C1—C14—C9	59.8 (2)
C7—C2—C3—C4	−0.7 (3)	C7—C8—C15—C16	−55.1 (2)
C1—C2—C3—C4	178.10 (18)	C9—C8—C15—C16	59.4 (2)
C2—C3—C4—C5	0.6 (3)	C8—C15—C16—C20	−123.56 (17)
C3—C4—C5—C6	0.3 (3)	C8—C15—C16—C17	117.18 (17)
C4—C5—C6—C7	−1.1 (3)	C8—C15—C16—C1	−2.4 (2)
C5—C6—C7—C2	1.0 (3)	C14—C1—C16—C20	66.91 (18)
C5—C6—C7—C8	−178.54 (18)	C2—C1—C16—C20	−178.79 (15)
C3—C2—C7—C6	−0.1 (3)	C14—C1—C16—C17	−176.91 (14)
C1—C2—C7—C6	−179.02 (17)	C2—C1—C16—C17	−62.61 (18)
C3—C2—C7—C8	179.51 (17)	C14—C1—C16—C15	−55.63 (19)
C1—C2—C7—C8	0.5 (2)	C2—C1—C16—C15	58.68 (19)
C6—C7—C8—C9	124.7 (2)	C20—C16—C17—O2	−131.2 (2)
C2—C7—C8—C9	−54.8 (2)	C15—C16—C17—O2	−8.8 (3)
C6—C7—C8—C15	−121.7 (2)	C1—C16—C17—O2	110.7 (2)
C2—C7—C8—C15	58.8 (2)	C20—C16—C17—O1	49.30 (19)
C7—C8—C9—C10	−126.4 (2)	C15—C16—C17—O1	171.68 (15)

C15—C8—C9—C10	118.7 (2)	C1—C16—C17—O1	−68.82 (19)
C7—C8—C9—C14	54.7 (2)	C17—C16—C20—O3	−109.9 (2)
C15—C8—C9—C14	−60.2 (2)	C15—C16—C20—O3	128.1 (2)
C14—C9—C10—C11	−0.3 (3)	C1—C16—C20—O3	7.6 (3)
C8—C9—C10—C11	−179.15 (19)	C17—C16—C20—O4	69.68 (19)
C9—C10—C11—C12	0.2 (3)	C15—C16—C20—O4	−52.4 (2)
C10—C11—C12—C13	0.0 (3)	C1—C16—C20—O4	−172.83 (15)
C11—C12—C13—C14	0.0 (3)	O2—C17—O1—C18	0.0 (3)
C12—C13—C14—C9	−0.2 (3)	C16—C17—O1—C18	179.50 (16)
C12—C13—C14—C1	179.54 (18)	C19—C18—O1—C17	−86.8 (2)
C10—C9—C14—C13	0.3 (3)	O3—C20—O4—C21	−3.8 (3)
C8—C9—C14—C13	179.30 (17)	C16—C20—O4—C21	176.68 (17)
C10—C9—C14—C1	−179.41 (17)	C22—C21—O4—C20	−121.4 (2)
C8—C9—C14—C1	−0.4 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O3^{i}	1.00	2.56	3.380 (2)	139

Symmetry code: (i) $x+1/2, -y+1/2, z+1/2$.