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Ethyl 4-(2,4-dichlorophenyl)-6-(6-methoxy-2-naphthyl)-2-oxocyclohex-3-ene-1-carboxylate

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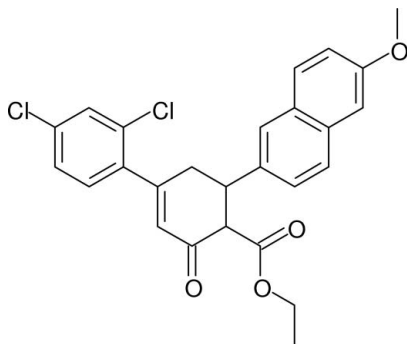
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.062; wR factor = 0.160; data-to-parameter ratio = 17.9.

In the title compound, $\text{C}_{26}\text{H}_{22}\text{Cl}_2\text{O}_4$, the cyclohexenone ring adopts an approximate half-chair conformation, with two C atoms displaced by -0.485 (6) and 0.218 (6) Å from the plane of the other four ring atoms. The dihedral angles between its four almost coplanar [maximum deviation = 0.006 (2) Å] atoms and the benzene and naphthalene ring systems are 59.26 (13) and 79.94 (9)°, respectively. The dihedral angle between the aromatic rings systems is 77.14 (7)°. A short intramolecular $\text{C}-\text{H}\cdots\text{Cl}$ contact generates an $S(6)$ ring. In the crystal, molecules are linked by $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{Cl}$ interactions to generate a three-dimensional network.

Related literature

For related structures and background references, see: Li *et al.* (2009a,b).



Experimental

Crystal data

$\text{C}_{26}\text{H}_{22}\text{Cl}_2\text{O}_4$
 $M_r = 469.34$
Monoclinic, $P2_1/c$
 $a = 14.2156$ (4) Å
 $b = 5.8647$ (2) Å
 $c = 27.3752$ (9) Å
 $\beta = 94.840$ (2)°
 $V = 2274.14$ (13) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.32$ mm⁻¹
 $T = 120$ K
 $0.20 \times 0.10 \times 0.07$ mm

Data collection

Nonius KappaCCD diffractometer
24499 measured reflections
5209 independent reflections
3171 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.081$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.160$
 $S = 1.05$
5209 reflections
291 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.39$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.32$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C6}-\text{H6}\cdots\text{O1}^{\text{i}}$	0.95	2.49	3.411 (4)	164
$\text{C8}-\text{H8A}\cdots\text{O4}^{\text{ii}}$	0.99	2.52	3.388 (4)	146
$\text{C8}-\text{H8B}\cdots\text{Cl2}$	0.99	2.69	3.365 (3)	125
$\text{C12}-\text{H12}\cdots\text{O1}^{\text{iii}}$	0.95	2.42	3.354 (4)	168
$\text{C14}-\text{H14}\cdots\text{O4}^{\text{ii}}$	0.95	2.33	3.270 (4)	170
$\text{C17}-\text{H17}\cdots\text{Cl1}^{\text{iv}}$	0.95	2.76	3.635 (3)	153

Symmetry codes: (i) $-x, -y + 2, -z$; (ii) $x, y + 1, z$; (iii) $-x, -y + 1, -z$; (iv) $x, -y + \frac{5}{2}, z + \frac{1}{2}$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor 1997), *SCALEPACK* and *SORTAV* (Blessing, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5016).

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supporting information

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Ethyl 4-(2,4-dichlorophenyl)-6-(6-methoxy-2-naphthyl)-2-oxocyclohex-3-ene-1-carboxylate

William T. A. Harrison, A. N. Mayekar, H. S. Yathirajan, B. Narayana and B. K. Sarojini

S1. Comment

The structure of the title compound, (I), (Fig. 1), was determined as part of our ongoing structural studies (Li *et al.*, 2009*a,b*) of substituted cyclohexenones.

The cyclohexenone ring (C7–C12) in (I) adopts an approximate half-chair conformation with C7/C8/C11/C12 statistically coplanar [r.m.s. deviation = 0.0004 Å; individual deviations = 0.0006 (19), -0.0003 (9), 0.0003 (9) and -0.006 (2) Å, respectively] and C9 and C10 displaced from their mean plane by -0.485 (6) and 0.218 (6) Å, respectively. Unlike the equivalent atoms in the related structures ethyl 6-(6-methoxy-2-naphthyl)-4-(4-methylphenyl)-2-oxocyclohex-3-ene-1-carboxylate, (II), (Li *et al.*, 2009*a*) and ethyl 6-(6-methoxy-2-naphthyl)-2-oxo-4-(2-thienyl)cyclohex-3-ene-1-carboxylate, (III), (Li *et al.*, 2009*b*), C9 and C10 in (I) do not display positional disorder. Both atoms are stereogenic centres: in the arbitrarily chosen asymmetric molecule, C9 has *R* configuration and C10 has *S*, but crystal symmetry generates a racemic mixture of enantiomers.

The dihedral angles between C7/C8/C11/C12 and the benzene (C1–C6) and naphthalene (C13–C22) ring systems are 59.26 (13) and 79.94 (9)°, respectively. The dihedral angle between the aromatic rings systems in (I) is 77.14 (7)°: equivalent values in (II) and (III) are 73.10 (5) and 86.04 (16)°, respectively. The naphthalene ring system (atoms C13–C22) in (I) shows rather high deviations from planarity: the r.m.s. deviation is 0.044 Å and maximum deviations are 0.074 (2) Å for C13 and -0.055 (2) for C21. If the two benzene rings (C13/C14/C15/C16/C21/C22 and C16–C21) are considered separately, their r.m.s. deviations are 0.018 and 0.007 Å, respectively, and the dihedral angle between them is 4.85 (16)°. Atom C23 of the terminal methyl group is displaced from the naphthalene ring by 0.466 (4) Å. A short intramolecular C8—H8B···C12 contact (Table 1) generates an S(6) ring.

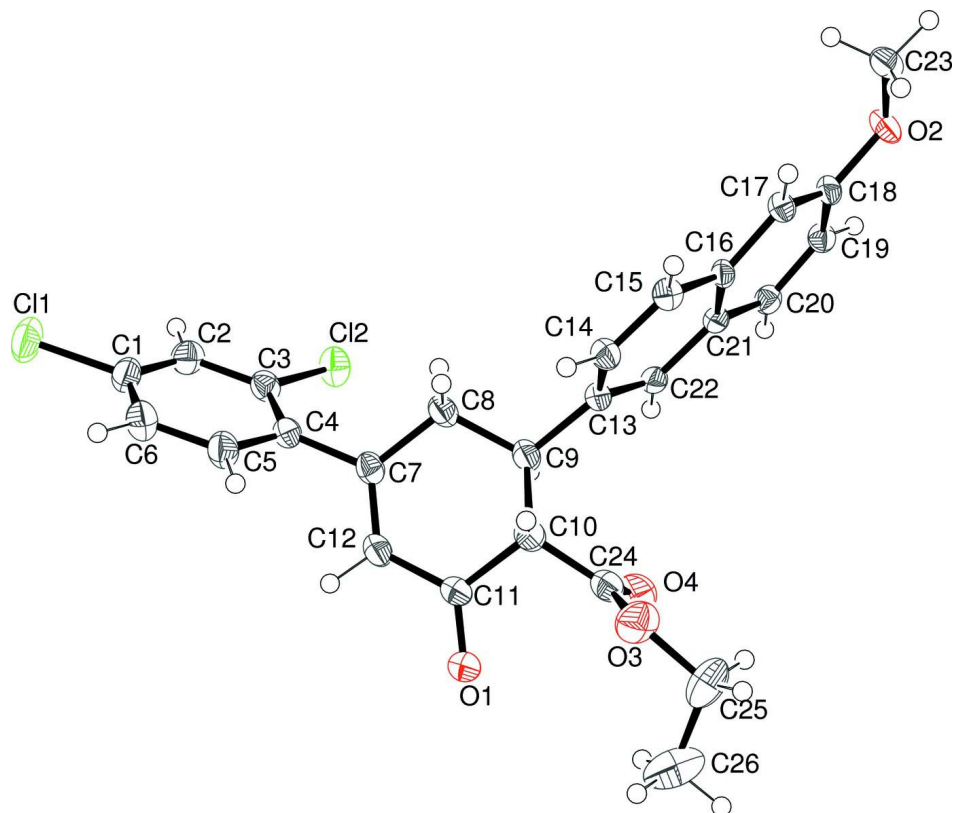
In the crystal, the molecules are linked by C—H···O and C—H···Cl interactions to generate a three-dimensional network.

S2. Experimental

(2E)-1-(2,4-Dichlorophenyl)-3-(6-methoxy-2-naphthyl)prop-2-en-1-one (1.8 g, 5 mmol) and ethyl acetoacetate (0.65 g, 5 mmol) were refluxed for 4 hr in 15 ml of ethanol in the presence of 0.8 ml 10% NaOH. The mixture was cooled to room temperature and the reaction mass was filtered and recrystallized using acetonitrile to yield colourless blocks of (I) (m.p.: 393–395 K).

S3. Refinement

The hydrogen atoms were geometrically placed (C—H = 0.95–1.00 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$. A rotating rigid-group model was applied to the methyl group.

**Figure 1**

View of the molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

Ethyl 4-(2,4-dichlorophenyl)-6-(6-methoxy-2-naphthyl)-2-oxocyclohex-3-ene-1-carboxylate

Crystal data

$C_{26}H_{22}Cl_2O_4$

$M_r = 469.34$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 14.2156\ (4)\ \text{\AA}$

$b = 5.8647\ (2)\ \text{\AA}$

$c = 27.3752\ (9)\ \text{\AA}$

$\beta = 94.840\ (2)^\circ$

$V = 2274.14\ (13)\ \text{\AA}^3$

$Z = 4$

$F(000) = 976$

$D_x = 1.371\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 19625 reflections

$\theta = 2.9\text{--}27.5^\circ$

$\mu = 0.32\ \text{mm}^{-1}$

$T = 120\ \text{K}$

Block, colourless

$0.20 \times 0.10 \times 0.07\ \text{mm}$

Data collection

Nonius KappaCCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω and φ scans

24499 measured reflections

5209 independent reflections

3171 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.081$

$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.1^\circ$

$h = -18 \rightarrow 18$

$k = -7 \rightarrow 7$

$l = -35 \rightarrow 35$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.160$
 $S = 1.05$
 5209 reflections
 291 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
 $w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 1.4128P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.39 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2743 (2)	1.2107 (5)	-0.05812 (11)	0.0325 (7)
C2	0.3364 (2)	1.0439 (5)	-0.04033 (11)	0.0317 (7)
H2	0.3962	1.0277	-0.0530	0.038*
C3	0.3101 (2)	0.9003 (5)	-0.00372 (10)	0.0271 (7)
C4	0.2229 (2)	0.9190 (5)	0.01610 (10)	0.0268 (7)
C5	0.1630 (2)	1.0922 (6)	-0.00319 (11)	0.0337 (8)
H5	0.1031	1.1100	0.0093	0.040*
C6	0.1876 (2)	1.2388 (6)	-0.03978 (12)	0.0364 (8)
H6	0.1457	1.3558	-0.0519	0.044*
C7	0.18917 (19)	0.7672 (5)	0.05408 (10)	0.0256 (7)
C8	0.2417 (2)	0.7606 (5)	0.10417 (10)	0.0279 (7)
H8A	0.2294	0.9038	0.1217	0.033*
H8B	0.3103	0.7529	0.1005	0.033*
C9	0.21375 (19)	0.5572 (5)	0.13528 (10)	0.0272 (7)
H9	0.2380	0.4157	0.1203	0.033*
C10	0.10628 (19)	0.5373 (6)	0.13368 (11)	0.0304 (7)
H10	0.0810	0.6780	0.1486	0.036*
C11	0.0638 (2)	0.5189 (6)	0.08069 (11)	0.0312 (7)
C12	0.1079 (2)	0.6548 (5)	0.04425 (11)	0.0311 (7)
H12	0.0774	0.6639	0.0121	0.037*
C13	0.26075 (19)	0.5753 (5)	0.18738 (10)	0.0256 (7)
C14	0.24668 (19)	0.7727 (5)	0.21574 (10)	0.0274 (7)
H14	0.2040	0.8869	0.2031	0.033*
C15	0.29374 (19)	0.8015 (5)	0.26125 (10)	0.0249 (6)
H15	0.2819	0.9335	0.2799	0.030*

C16	0.35953 (18)	0.6376 (5)	0.28076 (9)	0.0203 (6)
C17	0.41590 (18)	0.6728 (5)	0.32532 (10)	0.0220 (6)
H17	0.4088	0.8083	0.3437	0.026*
C18	0.48025 (19)	0.5124 (5)	0.34184 (10)	0.0232 (6)
C19	0.49063 (19)	0.3078 (5)	0.31567 (10)	0.0239 (6)
H19	0.5351	0.1965	0.3280	0.029*
C20	0.43709 (19)	0.2697 (5)	0.27280 (10)	0.0237 (6)
H20	0.4443	0.1311	0.2556	0.028*
C21	0.37076 (18)	0.4340 (5)	0.25358 (10)	0.0213 (6)
C22	0.31971 (19)	0.4083 (5)	0.20690 (10)	0.0235 (6)
H22	0.3267	0.2719	0.1889	0.028*
C23	0.5480 (2)	0.7485 (5)	0.40688 (11)	0.0318 (7)
H23A	0.5984	0.7451	0.4336	0.048*
H23B	0.4879	0.7852	0.4202	0.048*
H23C	0.5624	0.8647	0.3829	0.048*
C24	0.0723 (2)	0.3334 (6)	0.16028 (11)	0.0324 (7)
C25	-0.0480 (3)	0.1826 (8)	0.20559 (15)	0.0657 (12)
H25A	-0.0850	0.2342	0.2326	0.079*
H25B	0.0014	0.0764	0.2195	0.079*
C26	-0.1126 (3)	0.0587 (8)	0.16739 (18)	0.0768 (14)
H26A	-0.1457	-0.0640	0.1832	0.115*
H26B	-0.0750	-0.0062	0.1424	0.115*
H26C	-0.1588	0.1662	0.1519	0.115*
O1	-0.00458 (15)	0.3976 (4)	0.06997 (8)	0.0436 (6)
O2	0.54118 (13)	0.5310 (3)	0.38362 (7)	0.0301 (5)
O3	-0.00269 (15)	0.3819 (4)	0.18412 (9)	0.0468 (6)
O4	0.10805 (16)	0.1490 (4)	0.15843 (9)	0.0468 (6)
Cl1	0.30566 (6)	1.39074 (16)	-0.10447 (3)	0.0484 (3)
Cl2	0.38884 (5)	0.68373 (14)	0.01551 (3)	0.0360 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0409 (18)	0.0292 (18)	0.0274 (17)	-0.0051 (15)	0.0038 (14)	0.0047 (14)
C2	0.0321 (16)	0.0368 (19)	0.0272 (16)	-0.0022 (15)	0.0085 (13)	-0.0014 (15)
C3	0.0288 (15)	0.0312 (17)	0.0210 (15)	0.0002 (13)	0.0014 (12)	-0.0018 (13)
C4	0.0297 (15)	0.0310 (17)	0.0193 (15)	-0.0038 (14)	0.0005 (12)	0.0004 (13)
C5	0.0286 (16)	0.043 (2)	0.0297 (17)	0.0020 (15)	0.0037 (13)	0.0088 (15)
C6	0.0388 (18)	0.0384 (19)	0.0318 (18)	0.0057 (15)	0.0021 (15)	0.0072 (15)
C7	0.0262 (15)	0.0312 (17)	0.0195 (15)	0.0029 (13)	0.0025 (12)	0.0003 (13)
C8	0.0254 (15)	0.0380 (18)	0.0198 (15)	0.0001 (14)	-0.0003 (12)	0.0000 (13)
C9	0.0273 (15)	0.0326 (18)	0.0211 (15)	-0.0001 (13)	-0.0006 (12)	0.0021 (13)
C10	0.0255 (15)	0.0407 (19)	0.0247 (16)	-0.0023 (14)	0.0008 (13)	0.0023 (14)
C11	0.0257 (15)	0.0404 (19)	0.0265 (17)	-0.0059 (15)	-0.0041 (13)	0.0032 (15)
C12	0.0301 (16)	0.0409 (19)	0.0215 (15)	-0.0027 (15)	-0.0026 (13)	0.0041 (14)
C13	0.0266 (15)	0.0308 (17)	0.0190 (15)	-0.0062 (13)	0.0002 (12)	0.0028 (13)
C14	0.0251 (15)	0.0311 (17)	0.0254 (16)	0.0000 (13)	-0.0009 (13)	0.0041 (14)
C15	0.0249 (14)	0.0259 (16)	0.0241 (15)	0.0023 (13)	0.0036 (12)	-0.0004 (13)

C16	0.0198 (13)	0.0254 (16)	0.0157 (13)	-0.0001 (12)	0.0020 (11)	0.0037 (12)
C17	0.0241 (14)	0.0233 (15)	0.0186 (14)	0.0013 (12)	0.0015 (11)	-0.0012 (12)
C18	0.0245 (14)	0.0294 (16)	0.0156 (14)	-0.0020 (13)	0.0015 (11)	0.0011 (13)
C19	0.0260 (14)	0.0231 (16)	0.0226 (15)	0.0009 (13)	0.0017 (12)	0.0010 (13)
C20	0.0285 (15)	0.0214 (16)	0.0218 (15)	-0.0022 (12)	0.0051 (12)	-0.0011 (12)
C21	0.0210 (14)	0.0238 (16)	0.0194 (14)	-0.0037 (12)	0.0033 (11)	0.0010 (12)
C22	0.0262 (14)	0.0252 (16)	0.0194 (14)	-0.0062 (13)	0.0027 (12)	-0.0005 (13)
C23	0.0350 (17)	0.0341 (18)	0.0250 (16)	-0.0030 (14)	-0.0046 (13)	-0.0031 (14)
C24	0.0263 (16)	0.041 (2)	0.0288 (17)	-0.0013 (15)	-0.0035 (13)	0.0014 (16)
C25	0.062 (3)	0.081 (3)	0.058 (3)	-0.017 (2)	0.029 (2)	0.015 (2)
C26	0.055 (3)	0.074 (3)	0.104 (4)	-0.025 (2)	0.019 (3)	-0.006 (3)
O1	0.0360 (12)	0.0607 (16)	0.0325 (13)	-0.0196 (12)	-0.0070 (10)	0.0123 (12)
O2	0.0363 (12)	0.0296 (12)	0.0221 (11)	0.0050 (9)	-0.0103 (9)	-0.0021 (9)
O3	0.0386 (13)	0.0583 (16)	0.0455 (14)	-0.0024 (12)	0.0160 (11)	-0.0035 (13)
O4	0.0385 (13)	0.0447 (16)	0.0560 (16)	0.0066 (12)	-0.0034 (12)	0.0070 (13)
C11	0.0578 (6)	0.0480 (6)	0.0407 (5)	-0.0047 (4)	0.0109 (4)	0.0188 (4)
C12	0.0380 (4)	0.0395 (5)	0.0312 (4)	0.0087 (4)	0.0071 (3)	0.0055 (4)

Geometric parameters (Å, °)

C1—C2	1.378 (4)	C14—H14	0.9500
C1—C6	1.380 (4)	C15—C16	1.414 (4)
C1—C11	1.737 (3)	C15—H15	0.9500
C2—C3	1.384 (4)	C16—C17	1.417 (4)
C2—H2	0.9500	C16—C21	1.423 (4)
C3—C4	1.399 (4)	C17—C18	1.362 (4)
C3—C12	1.745 (3)	C17—H17	0.9500
C4—C5	1.400 (4)	C18—O2	1.379 (3)
C4—C7	1.479 (4)	C18—C19	1.412 (4)
C5—C6	1.387 (4)	C19—C20	1.362 (4)
C5—H5	0.9500	C19—H19	0.9500
C6—H6	0.9500	C20—C21	1.418 (4)
C7—C12	1.338 (4)	C20—H20	0.9500
C7—C8	1.506 (4)	C21—C22	1.423 (4)
C8—C9	1.537 (4)	C22—H22	0.9500
C8—H8A	0.9900	C23—O2	1.425 (3)
C8—H8B	0.9900	C23—H23A	0.9800
C9—C13	1.527 (4)	C23—H23B	0.9800
C9—C10	1.529 (4)	C23—H23C	0.9800
C9—H9	1.0000	C24—O4	1.198 (4)
C10—C24	1.501 (4)	C24—O3	1.328 (4)
C10—C11	1.528 (4)	C25—O3	1.480 (4)
C10—H10	1.0000	C25—C26	1.517 (6)
C11—O1	1.220 (3)	C25—H25A	0.9900
C11—C12	1.459 (4)	C25—H25B	0.9900
C12—H12	0.9500	C26—H26A	0.9800
C13—C22	1.368 (4)	C26—H26B	0.9800
C13—C14	1.417 (4)	C26—H26C	0.9800

C14—C15	1.374 (4)		
C2—C1—C6	121.4 (3)	C15—C14—H14	119.4
C2—C1—C11	119.6 (2)	C13—C14—H14	119.4
C6—C1—C11	119.0 (2)	C14—C15—C16	121.0 (3)
C1—C2—C3	118.9 (3)	C14—C15—H15	119.5
C1—C2—H2	120.5	C16—C15—H15	119.5
C3—C2—H2	120.5	C15—C16—C17	122.3 (3)
C2—C3—C4	122.4 (3)	C15—C16—C21	118.2 (2)
C2—C3—C12	117.1 (2)	C17—C16—C21	119.5 (2)
C4—C3—C12	120.5 (2)	C18—C17—C16	120.0 (3)
C3—C4—C5	116.2 (3)	C18—C17—H17	120.0
C3—C4—C7	125.2 (3)	C16—C17—H17	120.0
C5—C4—C7	118.6 (3)	C17—C18—O2	125.3 (3)
C6—C5—C4	122.7 (3)	C17—C18—C19	120.9 (2)
C6—C5—H5	118.7	O2—C18—C19	113.8 (2)
C4—C5—H5	118.7	C20—C19—C18	120.1 (3)
C1—C6—C5	118.4 (3)	C20—C19—H19	119.9
C1—C6—H6	120.8	C18—C19—H19	119.9
C5—C6—H6	120.8	C19—C20—C21	120.9 (3)
C12—C7—C4	118.7 (3)	C19—C20—H20	119.5
C12—C7—C8	121.6 (3)	C21—C20—H20	119.5
C4—C7—C8	119.3 (2)	C20—C21—C16	118.5 (2)
C7—C8—C9	113.1 (2)	C20—C21—C22	122.3 (3)
C7—C8—H8A	109.0	C16—C21—C22	119.2 (2)
C9—C8—H8A	109.0	C13—C22—C21	121.6 (3)
C7—C8—H8B	109.0	C13—C22—H22	119.2
C9—C8—H8B	109.0	C21—C22—H22	119.2
H8A—C8—H8B	107.8	O2—C23—H23A	109.5
C13—C9—C10	112.9 (2)	O2—C23—H23B	109.5
C13—C9—C8	110.5 (2)	H23A—C23—H23B	109.5
C10—C9—C8	110.4 (2)	O2—C23—H23C	109.5
C13—C9—H9	107.6	H23A—C23—H23C	109.5
C10—C9—H9	107.6	H23B—C23—H23C	109.5
C8—C9—H9	107.6	O4—C24—O3	125.1 (3)
C24—C10—C11	106.7 (2)	O4—C24—C10	123.0 (3)
C24—C10—C9	114.1 (2)	O3—C24—C10	111.9 (3)
C11—C10—C9	110.3 (2)	O3—C25—C26	111.3 (3)
C24—C10—H10	108.6	O3—C25—H25A	109.4
C11—C10—H10	108.6	C26—C25—H25A	109.4
C9—C10—H10	108.6	O3—C25—H25B	109.4
O1—C11—C12	122.2 (3)	C26—C25—H25B	109.4
O1—C11—C10	120.8 (3)	H25A—C25—H25B	108.0
C12—C11—C10	116.9 (2)	C25—C26—H26A	109.5
C7—C12—C11	123.0 (3)	C25—C26—H26B	109.5
C7—C12—H12	118.5	H26A—C26—H26B	109.5
C11—C12—H12	118.5	C25—C26—H26C	109.5
C22—C13—C14	118.7 (3)	H26A—C26—H26C	109.5

C22—C13—C9	121.4 (3)	H26B—C26—H26C	109.5
C14—C13—C9	119.8 (3)	C18—O2—C23	117.1 (2)
C15—C14—C13	121.1 (3)	C24—O3—C25	114.9 (3)
C6—C1—C2—C3	0.8 (5)	C8—C9—C13—C22	-120.9 (3)
C11—C1—C2—C3	-179.4 (2)	C10—C9—C13—C14	-67.7 (3)
C1—C2—C3—C4	-0.3 (4)	C8—C9—C13—C14	56.4 (3)
C1—C2—C3—C12	177.1 (2)	C22—C13—C14—C15	2.5 (4)
C2—C3—C4—C5	0.0 (4)	C9—C13—C14—C15	-174.9 (3)
C12—C3—C4—C5	-177.3 (2)	C13—C14—C15—C16	1.6 (4)
C2—C3—C4—C7	178.0 (3)	C14—C15—C16—C17	173.6 (3)
C12—C3—C4—C7	0.7 (4)	C14—C15—C16—C21	-4.4 (4)
C3—C4—C5—C6	-0.1 (5)	C15—C16—C17—C18	-178.1 (3)
C7—C4—C5—C6	-178.3 (3)	C21—C16—C17—C18	0.0 (4)
C2—C1—C6—C5	-1.0 (5)	C16—C17—C18—O2	177.6 (2)
C11—C1—C6—C5	179.2 (2)	C16—C17—C18—C19	-1.3 (4)
C4—C5—C6—C1	0.6 (5)	C17—C18—C19—C20	1.1 (4)
C3—C4—C7—C12	-122.5 (3)	O2—C18—C19—C20	-177.9 (2)
C5—C4—C7—C12	55.4 (4)	C18—C19—C20—C21	0.4 (4)
C3—C4—C7—C8	63.4 (4)	C19—C20—C21—C16	-1.7 (4)
C5—C4—C7—C8	-118.6 (3)	C19—C20—C21—C22	174.4 (3)
C12—C7—C8—C9	20.0 (4)	C15—C16—C21—C20	179.6 (2)
C4—C7—C8—C9	-166.2 (3)	C17—C16—C21—C20	1.4 (4)
C7—C8—C9—C13	-173.5 (2)	C15—C16—C21—C22	3.4 (4)
C7—C8—C9—C10	-47.9 (3)	C17—C16—C21—C22	-174.8 (2)
C13—C9—C10—C24	-59.8 (3)	C14—C13—C22—C21	-3.5 (4)
C8—C9—C10—C24	176.0 (3)	C9—C13—C22—C21	173.9 (2)
C13—C9—C10—C11	-179.8 (3)	C20—C21—C22—C13	-175.5 (3)
C8—C9—C10—C11	56.0 (3)	C16—C21—C22—C13	0.6 (4)
C24—C10—C11—O1	19.1 (4)	C11—C10—C24—O4	81.0 (4)
C9—C10—C11—O1	143.5 (3)	C9—C10—C24—O4	-41.0 (4)
C24—C10—C11—C12	-161.8 (3)	C11—C10—C24—O3	-96.8 (3)
C9—C10—C11—C12	-37.4 (4)	C9—C10—C24—O3	141.2 (3)
C4—C7—C12—C11	-173.8 (3)	C17—C18—O2—C23	-10.7 (4)
C8—C7—C12—C11	0.1 (5)	C19—C18—O2—C23	168.3 (2)
O1—C11—C12—C7	-171.8 (3)	O4—C24—O3—C25	-5.8 (5)
C10—C11—C12—C7	9.1 (5)	C10—C24—O3—C25	172.0 (3)
C10—C9—C13—C22	114.9 (3)	C26—C25—O3—C24	-81.2 (4)

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C6—H6 \cdots O1 ⁱ	0.95	2.49	3.411 (4)	164
C8—H8 <i>A</i> \cdots O4 ⁱⁱ	0.99	2.52	3.388 (4)	146
C8—H8 <i>B</i> \cdots C12	0.99	2.69	3.365 (3)	125
C12—H12 \cdots O1 ⁱⁱⁱ	0.95	2.42	3.354 (4)	168

C14—H14···O4 ⁱⁱ	0.95	2.33	3.270 (4)	170
C17—H17···C11 ^{iv}	0.95	2.76	3.635 (3)	153

Symmetry codes: (i) $-x, -y+2, -z$; (ii) $x, y+1, z$; (iii) $-x, -y+1, -z$; (iv) $x, -y+5/2, z+1/2$.