



Crystal structure of 8-[7,8-bis(4-chlorobenzoyl)-7*H*-cyclopenta[*a*]acenaphthylen-9-yl]naphthalene-1-carboxylic acid

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The title compound, C₄₀H₂₂Cl₂O₄, was formed by a Michael–Aldol domino reaction sequence, which coupled acenaphthenequinone with 4-chloroacetophenone in the presence of KOH in methanol. The dihedral angles between the central cyclopenta[*a*]acenaphthylene fused-ring system (r.m.s. deviation = 0.066 Å) and the 4-chlorobenzoyl rings are 62.25 (10) and 70.19 (10)°. The dihedral angle between the central ring system and the naphthoic acid grouping is 62.46 (7)°. This twisting of the pendant rings facilitates the formation of an intramolecular aromatic π – π stacking interaction between the 4-chlorobenzoyl and naphthoic acid rings, with centroid–centroid distances of 3.4533 (16) and 3.5311 (16) Å, and a C–H... π interaction between one of the H atoms of the central moiety and the 4-chlorobenzoyl ring with an H... π distance of 2.57 Å. In the crystal, carboxylic acid inversion dimers generate *R*₂²(8) loops. The dimers are linked by weak C–H...O and C–H...Cl hydrogen bonds and C–H... π interactions, generating a three-dimensional architecture.

1. Chemical context

Domino reactions (Sousa *et al.*, 2014; Kumar & Perumal 2014; Pokhodylo *et al.*, 2014; Feng *et al.* 2014; Ramachandran *et al.*, 2014; Basetti *et al.*, 2014), also called cascade or tandem reactions, are usually carried out to enable the efficient construction of complex molecules from simple substrates with high atom economy. In this reaction, multiple C–C or C–H bonds are formed in the same vessel, including different reaction mechanisms to form complex molecules without the purification of intermediates. These reactions are often used in medical or combinatorial chemistry to synthesize complex active drug molecules (Sudhapriya *et al.*, 2014; Tietze *et al.*, 2014; Fu *et al.*, 2013; Shestopalov *et al.*, 2013; Zohreh & Alizadeh, 2013; Renault *et al.*, 2007). Domino reactions are classified as homo-domino processes and hetero-domino processes (Nesi *et al.*, 1999).

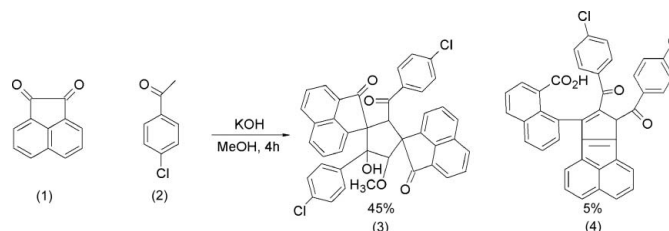
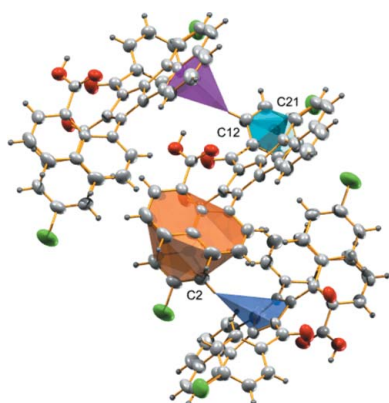
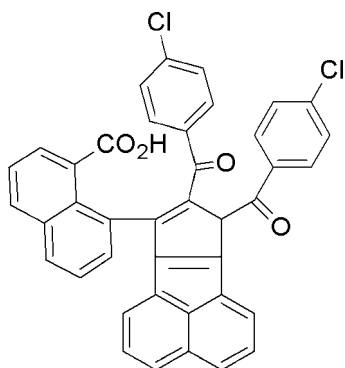


Figure 1
Reaction scheme showing the synthesis of the title compound (4).

One of the attractive strategies for constructing complex molecules (Filippini *et al.*, 1995; List *et al.*, 2000; Wang *et al.*, 2007) is a domino sequence of Michael addition and aldol condensation. In this article, we report the formation of the title compound (4) through a domino reaction sequence involving Claisen–Schmidt condensation and benzil–benzilic acid rearrangement between acenaphthenequinone (1) and 4-chloroacetophenone (2) in the presence of methanolic KOH (Fig. 1).



2. Structural commentary

In the title compound, the 4-chlorobenzoyl units are approximately coplanar with slight twisting [dihedral angle, 18.49 (13)°] and nearly parallel to the plane of naphthoic acid moiety with dihedral angles of 8.82 (11) and 12.06 (11)°. The C=O oxygen atoms of the two 4-chlorobenzoyl moieties point toward each other. The central cyclopenta[acenaphthylene] ring system makes dihedral angles of 62.25 (10) and 70.19 (10)° with the 4-chlorobenzoyl units and 62.46 (7)° with the naphthoic acid grouping. This twisting minimizes steric interactions among the substituents (Fig. 2) and facilitates the formation of intramolecular π – π interactions between the 4-chlorobenzoyl and naphthoic acid rings with centroid centroid distances of 3.4533 (16) and 3.5311 (16) Å and a C–H... π interaction between one of the hydrogen atoms of the central moiety and the 4-chlorobenzoyl ring.

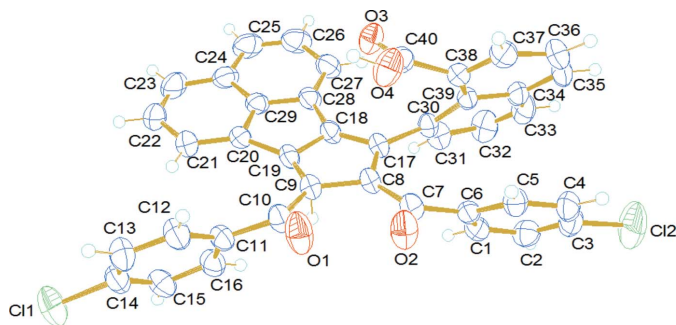


Figure 2

ORTEP view of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

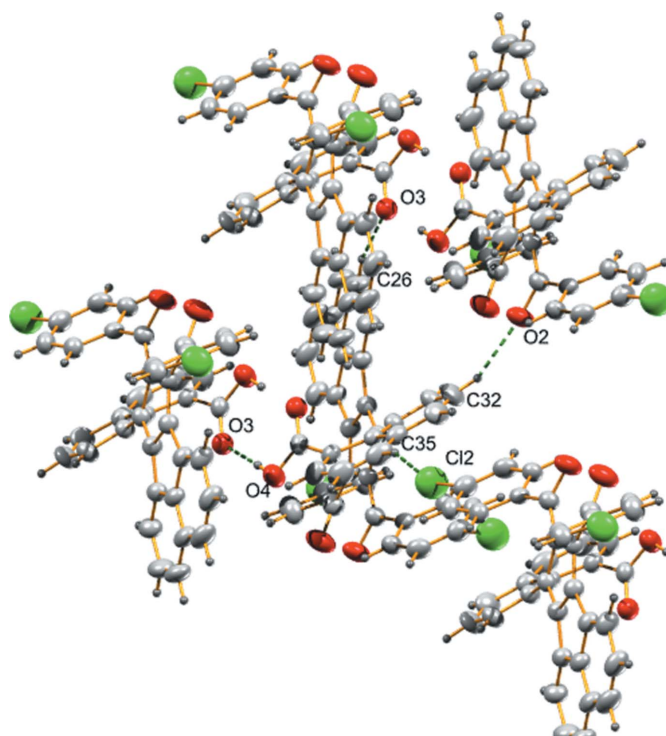


Figure 3

Hydrogen-bonding interactions (dashed lines) in the title compound.

3. Supramolecular features

There are four intermolecular hydrogen-bonding interactions present in the crystal. The carbonyl oxygen atoms (O2 and O3) accept three hydrogen bonds; one with the hydrogen atom from a carboxylic acid group of a neighboring molecule with $D\cdots A$ distance of 2.649 (3) Å ($-x, 1 - y, 2 - z$) and the other two with the hydrogen atoms attached to atoms C32 and C26 of the naphthoic acid and cyclopenta[acenaphthylene] rings, respectively, of adjacent molecules with $D\cdots A$ distances of 3.301 (4) ($1 + x, y, z$) and 3.416 (4) Å ($1 - x, 1 - y, 2 - z$) (Fig. 3). The fourth interaction is between the H atom attached to the naphthoic acid ring and a chlorine atom of the 4-chlorobenzoyl moiety with a $D\cdots A$ distance of 3.619 (3) Å ($1 - x, -y, 3 - z$). Furthermore, there are two C–H... π interactions found between hydrogen atoms (H2 and H12)

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C18–C20/C28/C29 ring, Cg2 is the centroid of the C24–C29 ring and Cg3 is the centroid of the C11–C16 ring.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------------|----------|-------------|-------------|---------------|
| O4–H4 ⁱ ...O3 ⁱ | 0.84 (1) | 1.81 (1) | 2.649 (3) | 178 (4) |
| C26–H26...O3 ⁱⁱ | 0.93 | 2.52 | 3.416 (4) | 163 |
| C32–H32...O2 ⁱⁱⁱ | 0.93 | 2.47 | 3.301 (4) | 149 |
| C35–H35...Cl2 ^{iv} | 0.93 | 2.74 | 3.619 (3) | 157 |
| C2–H2...Cg1 ^v | 0.93 | 2.87 | 3.577 (3) | 134 |
| C12–H12...Cg2 ^{vi} | 0.93 | 2.84 | 3.725 (3) | 160 |
| C21–H21...Cg3 | 0.93 | 2.57 | 3.425 (3) | 152 |

Symmetry codes: (i) $-x, -y + 1, -z + 2$; (ii) $-x + 1, -y + 1, -z + 2$; (iii) $x + 1, y, z$; (iv) $-x + 1, -y, -z + 3$; (v) $-x + 1, -y, -z + 2$; (vi) $x - 1, y, z$.

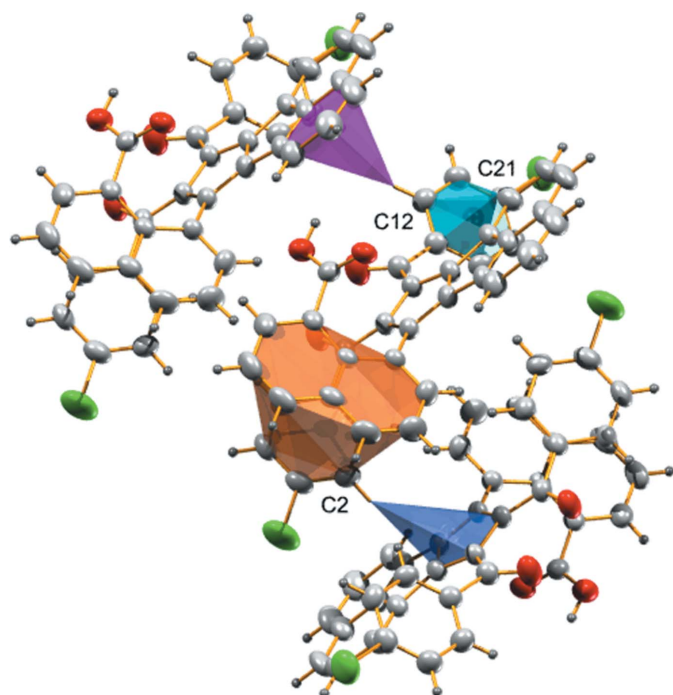


Figure 4
C—H $\cdots\pi$ and π — π interactions found in the title compound.

and the five- and six-membered rings of the cyclopenta[*a*]acenaphthylene and 4-chlorobenzoyl moieties of neighbouring molecules (Fig. 4), with H $\cdots\pi$ distances of 2.87 and 2.84 Å (Table 1).

The packing appears to be controlled by classical and non-classical hydrogen bonds and three C—H $\cdots\pi$ interactions (Mathew *et al.*, 2013). Fig. 5 shows the packing of the title compound viewed along the *a* axis.

4. Synthesis and crystallization

A mixture of acenaphthenequinone (1) (4.6 g, 25 mmol), 4-chloroacetophenone (2) (4.2 g, 27 mmol) and powdered potassium hydroxide (1.0 g) in methanol (30 ml) was stirred around 333 K for 4 h and later kept in a refrigerator for 48 h. The reaction mixture was concentrated and the residue was chromatographed over silica gel. Product (3) was obtained (Vadakkan *et al.*, 2003) by elution with a mixture (9:1) of hexane and ethyl acetate. Elution with a mixture of (1:1) methanol and ethyl acetate yielded the product (4) (Fig. 1). Red blocks of compound (4) were recrystallized from a solvent mixture of ethyl acetate and dichloromethane.

Yield 0.8 g (5%); m.p. >523 K; IR (KBr, ν_{\max}): 3370 (OH), 1732 (C=O) cm^{-1} ; ^1H NMR (CDCl_3): δ 8.00–5.30 (*m*, 20H, aromatic); ^{13}C NMR (CDCl_3): δ 207.57, 190.82, 179.39, 138.71, 135.57, 134.23, 134.17, 133.77, 132.57, 131.94, 131.69, 131.31, 130.40, 130.29, 129.90, 129.58, 129.22, 128.90, 128.85, 128.42, 128.06, 127.74, 127.66, 127.23, 126.54, 125.76, 125.64, 124.94, 124.38, 119.77, 103.38, 70.96; MS: *m/z* 636 (M^+); Analysis calculated for $\text{C}_{40}\text{H}_{22}\text{Cl}_2\text{O}_4$: C: 75.36, H: 3.48; found: C: 75.26, H: 3.30.

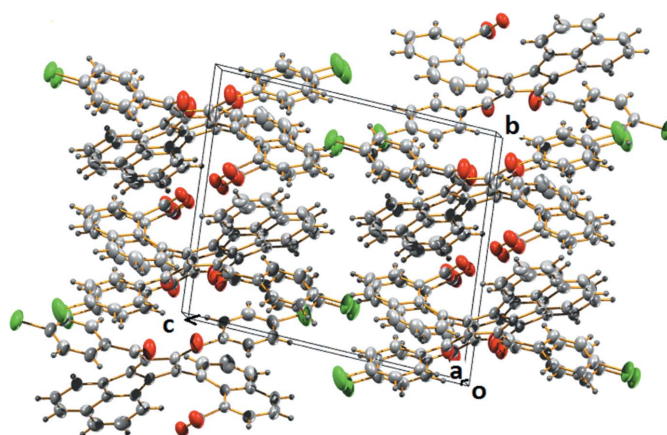


Figure 5
A packing diagram of the title compound viewed along the *a* axis.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms on C were placed in calculated positions, guided by difference maps, with C—H bond distances of 0.93 Å. H atoms were assigned as $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Hydrogen atom H4' of the naphthoic acid group was located from a difference Fourier map and refined with a distance restraint of O—H = 0.84 (1) Å. The low-angle

Table 2
Experimental details.

| | |
|--|--|
| Crystal data | |
| Chemical formula | $\text{C}_{40}\text{H}_{22}\text{Cl}_2\text{O}_4$ |
| M_r | 637.47 |
| Crystal system, space group | Triclinic, $P\bar{1}$ |
| Temperature (K) | 296 |
| <i>a</i> , <i>b</i> , <i>c</i> (Å) | 9.1617 (6), 12.5518 (8), 13.9305 (8) |
| α , β , γ (°) | 84.669 (3), 88.468 (3), 72.364 (3) |
| <i>V</i> (Å ³) | 1520.05 (17) |
| <i>Z</i> | 2 |
| Radiation type | Mo <i>K</i> α |
| μ (mm ⁻¹) | 0.26 |
| Crystal size (mm) | 0.35 × 0.30 × 0.25 |
| Data collection | |
| Diffractometer | Bruker Kappa APEXII CCD |
| Absorption correction | Multi-scan (<i>SADABS</i> ; Bruker, 2004) |
| T_{\min} , T_{\max} | 0.891, 0.908 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 19996, 5287, 4251 |
| R_{int} | 0.033 |
| ($\sin \theta/\lambda$) _{max} (Å ⁻¹) | 0.595 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, <i>S</i> | 0.052, 0.152, 1.12 |
| No. of reflections | 5287 |
| No. of parameters | 419 |
| No. of restraints | 1 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³) | 0.51, −0.78 |

Computer programs: *APEX2*, *SAINT* and *XPREP* (Bruker, 2004), *SHELXS97*, *SHELXL97* and *SHELXL2014* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg, 2010), and *publCIF* (Westrip, 2010).

reflections (001), ($\bar{1}01$) and ($0\bar{1}1$) were omitted from the refinement owing to bad agreement.

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Crystal structure of 8-[7,8-bis(4-chlorobenzoyl)-7H-cyclopenta[a]acenaphthylen-9-yl]naphthalene-1-carboxylic acid

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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: *SHELXL2014* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2010); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *pubCIF* (Westrip, 2010).

8-[7,8-Bis(4-chlorobenzoyl)-7H-cyclopenta[a]acenaphthylen-9-yl]naphthalene-1-carboxylic acid

Crystal data

$C_{40}H_{22}Cl_2O_4$

$M_r = 637.47$

Triclinic, $P\bar{1}$

$a = 9.1617$ (6) Å

$b = 12.5518$ (8) Å

$c = 13.9305$ (8) Å

$\alpha = 84.669$ (3)°

$\beta = 88.468$ (3)°

$\gamma = 72.364$ (3)°

$V = 1520.05$ (17) Å³

$Z = 2$

$F(000) = 656$

$D_x = 1.393$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9963 reflections

$\theta = 2.4$ – 28.1 °

$\mu = 0.26$ mm⁻¹

$T = 296$ K

Block, red

$0.35 \times 0.30 \times 0.25$ mm

Data collection

Bruker axs kappa apex2 CCD Diffractometer

ω and ϕ scan

Absorption correction: multi-scan

(*SADABS*; Bruker, 2004)

$T_{\min} = 0.891$, $T_{\max} = 0.908$

19996 measured reflections

5287 independent reflections

4251 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 25.0$ °, $\theta_{\min} = 2.2$ °

$h = -10 \rightarrow 10$

$k = -14 \rightarrow 14$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.055$

$wR(F^2) = 0.154$

$S = 1.16$

5287 reflections

419 parameters

1 restraint

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 1.3725P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.51$ e Å⁻³

$\Delta\rho_{\min} = -0.78$ e Å⁻³

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|--------------|----------------------------------|
| C1 | 0.3710 (3) | −0.0055 (2) | 1.14118 (19) | 0.0423 (6) |
| H1 | 0.4150 | −0.0104 | 1.0802 | 0.051* |
| C2 | 0.4574 (3) | −0.0622 (2) | 1.2201 (2) | 0.0494 (7) |
| H2 | 0.5584 | −0.1061 | 1.2129 | 0.059* |
| C3 | 0.3904 (4) | −0.0521 (3) | 1.3095 (2) | 0.0534 (7) |
| C4 | 0.2397 (4) | 0.0087 (2) | 1.32183 (19) | 0.0497 (7) |
| H4 | 0.1965 | 0.0132 | 1.3831 | 0.060* |
| C5 | 0.1536 (3) | 0.0630 (2) | 1.24211 (18) | 0.0410 (6) |
| H5 | 0.0508 | 0.1027 | 1.2492 | 0.049* |
| C6 | 0.2204 (3) | 0.0584 (2) | 1.15107 (17) | 0.0346 (5) |
| C7 | 0.1283 (3) | 0.1184 (2) | 1.06661 (18) | 0.0389 (6) |
| C8 | 0.1805 (3) | 0.1835 (2) | 0.99406 (16) | 0.0354 (5) |
| C9 | 0.1165 (3) | 0.2180 (2) | 0.89523 (16) | 0.0355 (5) |
| H9 | 0.1824 | 0.1478 | 0.8726 | 0.043* |
| C10 | −0.0153 (3) | 0.1981 (2) | 0.85791 (18) | 0.0429 (6) |
| C11 | −0.0373 (3) | 0.2001 (2) | 0.75185 (18) | 0.0395 (6) |
| C12 | −0.1801 (3) | 0.2556 (2) | 0.7114 (2) | 0.0491 (7) |
| H12 | −0.2595 | 0.2938 | 0.7501 | 0.059* |
| C13 | −0.2052 (3) | 0.2546 (3) | 0.6143 (2) | 0.0525 (7) |
| H13 | −0.2998 | 0.2939 | 0.5867 | 0.063* |
| C14 | −0.0878 (3) | 0.1946 (2) | 0.55900 (19) | 0.0476 (7) |
| C15 | 0.0539 (3) | 0.1368 (3) | 0.5976 (2) | 0.0494 (7) |
| H15 | 0.1315 | 0.0958 | 0.5592 | 0.059* |
| C16 | 0.0788 (3) | 0.1407 (2) | 0.6946 (2) | 0.0458 (6) |
| H16 | 0.1745 | 0.1031 | 0.7215 | 0.055* |
| C17 | 0.3071 (3) | 0.2282 (2) | 1.00154 (16) | 0.0356 (5) |
| C18 | 0.3212 (3) | 0.2850 (2) | 0.91319 (17) | 0.0370 (6) |
| C19 | 0.2079 (3) | 0.2776 (2) | 0.84755 (16) | 0.0369 (6) |
| C20 | 0.2216 (3) | 0.3468 (2) | 0.75736 (17) | 0.0395 (6) |
| C21 | 0.1484 (4) | 0.3781 (2) | 0.66922 (19) | 0.0513 (7) |
| H21 | 0.0674 | 0.3521 | 0.6547 | 0.062* |
| C22 | 0.1988 (4) | 0.4506 (3) | 0.6011 (2) | 0.0585 (8) |
| H22 | 0.1514 | 0.4695 | 0.5411 | 0.070* |
| C23 | 0.3133 (4) | 0.4933 (3) | 0.6199 (2) | 0.0620 (9) |
| H23 | 0.3423 | 0.5405 | 0.5729 | 0.074* |
| C24 | 0.3892 (4) | 0.4669 (2) | 0.7103 (2) | 0.0500 (7) |
| C25 | 0.5021 (4) | 0.5104 (3) | 0.7430 (3) | 0.0631 (9) |
| H25 | 0.5372 | 0.5603 | 0.7022 | 0.076* |
| C26 | 0.5604 (4) | 0.4798 (3) | 0.8343 (3) | 0.0616 (8) |

| | | | | |
|-----|---------------|---------------|--------------|-------------|
| H26 | 0.6338 | 0.5104 | 0.8544 | 0.074* |
| C27 | 0.5130 (3) | 0.4036 (2) | 0.8989 (2) | 0.0491 (7) |
| H27 | 0.5551 | 0.3837 | 0.9604 | 0.059* |
| C28 | 0.4040 (3) | 0.3594 (2) | 0.86951 (17) | 0.0390 (6) |
| C29 | 0.3416 (3) | 0.3925 (2) | 0.77553 (18) | 0.0410 (6) |
| C30 | 0.4027 (3) | 0.2227 (2) | 1.08743 (17) | 0.0359 (5) |
| C31 | 0.5582 (3) | 0.1748 (3) | 1.0823 (2) | 0.0483 (7) |
| H31 | 0.6034 | 0.1592 | 1.0226 | 0.058* |
| C32 | 0.6509 (3) | 0.1488 (3) | 1.1650 (2) | 0.0596 (8) |
| H32 | 0.7566 | 0.1189 | 1.1595 | 0.072* |
| C33 | 0.5861 (3) | 0.1672 (3) | 1.2529 (2) | 0.0560 (8) |
| H33 | 0.6466 | 0.1433 | 1.3080 | 0.067* |
| C34 | 0.4282 (3) | 0.2221 (2) | 1.26200 (18) | 0.0424 (6) |
| C35 | 0.3591 (4) | 0.2439 (3) | 1.35317 (19) | 0.0553 (8) |
| H35 | 0.4190 | 0.2199 | 1.4085 | 0.066* |
| C36 | 0.2086 (4) | 0.2987 (3) | 1.3618 (2) | 0.0601 (9) |
| H36 | 0.1642 | 0.3073 | 1.4225 | 0.072* |
| C37 | 0.1190 (4) | 0.3425 (2) | 1.2789 (2) | 0.0529 (7) |
| H37 | 0.0163 | 0.3830 | 1.2850 | 0.063* |
| C38 | 0.1818 (3) | 0.3261 (2) | 1.18879 (17) | 0.0376 (6) |
| C39 | 0.3359 (3) | 0.2582 (2) | 1.17763 (16) | 0.0337 (5) |
| C40 | 0.0952 (3) | 0.3994 (2) | 1.10642 (19) | 0.0402 (6) |
| O1 | -0.1211 (2) | 0.1783 (2) | 0.90948 (15) | 0.0642 (6) |
| O2 | -0.0068 (2) | 0.1055 (2) | 1.06624 (14) | 0.0571 (6) |
| O3 | 0.1576 (2) | 0.45386 (16) | 1.05156 (14) | 0.0489 (5) |
| O4 | -0.0494 (2) | 0.40610 (19) | 1.10215 (16) | 0.0585 (6) |
| Cl1 | -0.12238 (12) | 0.18916 (9) | 0.43778 (6) | 0.0775 (3) |
| Cl2 | 0.50109 (14) | -0.11781 (11) | 1.40953 (8) | 0.1010 (4) |
| H4' | -0.082 (5) | 0.451 (3) | 1.053 (2) | 0.100 (15)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0453 (15) | 0.0426 (14) | 0.0383 (14) | -0.0109 (12) | 0.0040 (11) | -0.0090 (11) |
| C2 | 0.0440 (16) | 0.0444 (15) | 0.0557 (17) | -0.0074 (13) | -0.0055 (13) | -0.0022 (13) |
| C3 | 0.062 (2) | 0.0541 (17) | 0.0449 (16) | -0.0218 (15) | -0.0142 (14) | 0.0106 (13) |
| C4 | 0.0630 (19) | 0.0581 (17) | 0.0326 (14) | -0.0273 (15) | 0.0041 (13) | 0.0017 (12) |
| C5 | 0.0403 (14) | 0.0481 (15) | 0.0349 (13) | -0.0148 (12) | 0.0042 (11) | -0.0010 (11) |
| C6 | 0.0394 (14) | 0.0367 (13) | 0.0318 (12) | -0.0176 (11) | 0.0012 (10) | -0.0030 (10) |
| C7 | 0.0382 (14) | 0.0475 (15) | 0.0328 (13) | -0.0149 (12) | 0.0010 (10) | -0.0056 (11) |
| C8 | 0.0358 (13) | 0.0457 (14) | 0.0264 (11) | -0.0139 (11) | -0.0019 (10) | -0.0060 (10) |
| C9 | 0.0369 (13) | 0.0408 (13) | 0.0289 (12) | -0.0116 (11) | -0.0029 (10) | -0.0040 (10) |
| C10 | 0.0442 (15) | 0.0511 (15) | 0.0349 (13) | -0.0162 (13) | -0.0055 (11) | -0.0041 (11) |
| C11 | 0.0399 (14) | 0.0447 (14) | 0.0375 (13) | -0.0169 (12) | -0.0076 (11) | -0.0053 (11) |
| C12 | 0.0461 (16) | 0.0570 (17) | 0.0407 (15) | -0.0079 (13) | -0.0055 (12) | -0.0112 (13) |
| C13 | 0.0477 (17) | 0.0632 (18) | 0.0433 (15) | -0.0114 (14) | -0.0118 (13) | -0.0026 (13) |
| C14 | 0.0561 (18) | 0.0574 (17) | 0.0345 (14) | -0.0233 (14) | -0.0061 (12) | -0.0076 (12) |
| C15 | 0.0497 (17) | 0.0569 (17) | 0.0436 (15) | -0.0156 (14) | 0.0018 (13) | -0.0169 (13) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C16 | 0.0395 (15) | 0.0504 (16) | 0.0471 (15) | -0.0114 (13) | -0.0096 (12) | -0.0063 (12) |
| C17 | 0.0381 (14) | 0.0432 (13) | 0.0262 (11) | -0.0121 (11) | -0.0011 (10) | -0.0062 (10) |
| C18 | 0.0424 (14) | 0.0433 (14) | 0.0274 (12) | -0.0146 (11) | 0.0014 (10) | -0.0081 (10) |
| C19 | 0.0452 (15) | 0.0424 (14) | 0.0249 (11) | -0.0146 (12) | -0.0007 (10) | -0.0065 (10) |
| C20 | 0.0532 (16) | 0.0360 (13) | 0.0300 (12) | -0.0139 (12) | 0.0002 (11) | -0.0056 (10) |
| C21 | 0.071 (2) | 0.0480 (16) | 0.0335 (14) | -0.0162 (15) | -0.0075 (13) | -0.0021 (12) |
| C22 | 0.087 (2) | 0.0489 (17) | 0.0355 (15) | -0.0166 (17) | -0.0058 (15) | 0.0035 (12) |
| C23 | 0.093 (3) | 0.0475 (17) | 0.0427 (16) | -0.0210 (17) | 0.0091 (16) | 0.0076 (13) |
| C24 | 0.069 (2) | 0.0363 (14) | 0.0459 (16) | -0.0185 (14) | 0.0091 (14) | -0.0044 (12) |
| C25 | 0.080 (2) | 0.0496 (17) | 0.068 (2) | -0.0344 (17) | 0.0146 (18) | -0.0007 (15) |
| C26 | 0.069 (2) | 0.0560 (18) | 0.073 (2) | -0.0374 (17) | 0.0065 (17) | -0.0108 (16) |
| C27 | 0.0566 (18) | 0.0527 (16) | 0.0451 (15) | -0.0253 (14) | 0.0013 (13) | -0.0109 (13) |
| C28 | 0.0458 (15) | 0.0416 (14) | 0.0324 (13) | -0.0164 (12) | 0.0048 (11) | -0.0083 (10) |
| C29 | 0.0531 (16) | 0.0366 (13) | 0.0353 (13) | -0.0157 (12) | 0.0069 (11) | -0.0074 (10) |
| C30 | 0.0350 (13) | 0.0437 (14) | 0.0303 (12) | -0.0138 (11) | -0.0048 (10) | -0.0015 (10) |
| C31 | 0.0374 (15) | 0.0632 (18) | 0.0455 (15) | -0.0170 (13) | 0.0016 (12) | -0.0058 (13) |
| C32 | 0.0335 (15) | 0.079 (2) | 0.065 (2) | -0.0185 (15) | -0.0123 (14) | 0.0072 (17) |
| C33 | 0.0492 (18) | 0.069 (2) | 0.0524 (18) | -0.0242 (15) | -0.0258 (14) | 0.0110 (15) |
| C34 | 0.0545 (17) | 0.0419 (14) | 0.0343 (13) | -0.0204 (13) | -0.0119 (12) | 0.0024 (11) |
| C35 | 0.085 (2) | 0.0581 (18) | 0.0294 (14) | -0.0312 (18) | -0.0151 (14) | 0.0025 (12) |
| C36 | 0.093 (3) | 0.0601 (19) | 0.0286 (14) | -0.0247 (19) | 0.0057 (15) | -0.0093 (13) |
| C37 | 0.067 (2) | 0.0442 (15) | 0.0418 (15) | -0.0078 (14) | 0.0091 (14) | -0.0083 (12) |
| C38 | 0.0475 (15) | 0.0338 (13) | 0.0317 (12) | -0.0121 (11) | -0.0002 (11) | -0.0038 (10) |
| C39 | 0.0401 (14) | 0.0356 (12) | 0.0281 (12) | -0.0157 (11) | -0.0053 (10) | -0.0005 (9) |
| C40 | 0.0396 (15) | 0.0392 (14) | 0.0385 (14) | -0.0064 (11) | -0.0022 (11) | -0.0046 (11) |
| O1 | 0.0481 (12) | 0.1076 (19) | 0.0438 (11) | -0.0367 (13) | -0.0076 (9) | 0.0055 (11) |
| O2 | 0.0446 (12) | 0.0895 (16) | 0.0437 (11) | -0.0339 (11) | -0.0035 (9) | 0.0091 (10) |
| O3 | 0.0452 (11) | 0.0517 (11) | 0.0462 (11) | -0.0126 (9) | -0.0090 (9) | 0.0099 (9) |
| O4 | 0.0407 (12) | 0.0666 (14) | 0.0605 (14) | -0.0107 (10) | -0.0047 (10) | 0.0153 (11) |
| Cl1 | 0.0868 (7) | 0.1123 (8) | 0.0380 (4) | -0.0333 (6) | -0.0096 (4) | -0.0165 (4) |
| Cl2 | 0.0940 (8) | 0.1317 (10) | 0.0678 (6) | -0.0311 (7) | -0.0373 (6) | 0.0394 (6) |

Geometric parameters (Å, °)

| | | | |
|--------|-----------|---------|-----------|
| C1—C2 | 1.379 (4) | C20—C29 | 1.423 (4) |
| C1—C6 | 1.380 (4) | C21—C22 | 1.418 (4) |
| C1—H1 | 0.9300 | C21—H21 | 0.9300 |
| C2—C3 | 1.373 (4) | C22—C23 | 1.355 (5) |
| C2—H2 | 0.9300 | C22—H22 | 0.9300 |
| C3—C4 | 1.376 (4) | C23—C24 | 1.418 (4) |
| C3—Cl2 | 1.734 (3) | C23—H23 | 0.9300 |
| C4—C5 | 1.378 (4) | C24—C29 | 1.398 (4) |
| C4—H4 | 0.9300 | C24—C25 | 1.411 (5) |
| C5—C6 | 1.392 (3) | C25—C26 | 1.368 (5) |
| C5—H5 | 0.9300 | C25—H25 | 0.9300 |
| C6—C7 | 1.472 (3) | C26—C27 | 1.408 (4) |
| C7—O2 | 1.297 (3) | C26—H26 | 0.9300 |
| C7—C8 | 1.405 (3) | C27—C28 | 1.369 (4) |

| | | | |
|-----------|-----------|-------------|------------|
| C8—C17 | 1.443 (3) | C27—H27 | 0.9300 |
| C8—C9 | 1.485 (3) | C28—C29 | 1.417 (4) |
| C9—C19 | 1.399 (3) | C30—C31 | 1.372 (4) |
| C9—C10 | 1.425 (4) | C30—C39 | 1.431 (3) |
| C9—H9 | 0.9800 | C31—C32 | 1.403 (4) |
| C10—O1 | 1.262 (3) | C31—H31 | 0.9300 |
| C10—C11 | 1.493 (3) | C32—C33 | 1.355 (5) |
| C11—C16 | 1.381 (4) | C32—H32 | 0.9300 |
| C11—C12 | 1.389 (4) | C33—C34 | 1.409 (4) |
| C12—C13 | 1.381 (4) | C33—H33 | 0.9300 |
| C12—H12 | 0.9300 | C34—C35 | 1.416 (4) |
| C13—C14 | 1.375 (4) | C34—C39 | 1.423 (3) |
| C13—H13 | 0.9300 | C35—C36 | 1.350 (5) |
| C14—C15 | 1.377 (4) | C35—H35 | 0.9300 |
| C14—C11 | 1.738 (3) | C36—C37 | 1.403 (4) |
| C15—C16 | 1.385 (4) | C36—H36 | 0.9300 |
| C15—H15 | 0.9300 | C37—C38 | 1.373 (4) |
| C16—H16 | 0.9300 | C37—H37 | 0.9300 |
| C17—C18 | 1.387 (3) | C38—C39 | 1.424 (4) |
| C17—C30 | 1.486 (3) | C38—C40 | 1.484 (4) |
| C18—C19 | 1.431 (3) | C40—O3 | 1.222 (3) |
| C18—C28 | 1.455 (4) | C40—O4 | 1.305 (3) |
| C19—C20 | 1.485 (3) | O4—H4' | 0.842 (10) |
| C20—C21 | 1.381 (4) | | |
| | | | |
| C2—C1—C6 | 121.2 (3) | C29—C20—C19 | 105.0 (2) |
| C2—C1—H1 | 119.4 | C20—C21—C22 | 118.9 (3) |
| C6—C1—H1 | 119.4 | C20—C21—H21 | 120.6 |
| C3—C2—C1 | 118.2 (3) | C22—C21—H21 | 120.6 |
| C3—C2—H2 | 120.9 | C23—C22—C21 | 122.8 (3) |
| C1—C2—H2 | 120.9 | C23—C22—H22 | 118.6 |
| C2—C3—C4 | 122.1 (3) | C21—C22—H22 | 118.6 |
| C2—C3—C12 | 118.4 (3) | C22—C23—C24 | 120.7 (3) |
| C4—C3—C12 | 119.4 (2) | C22—C23—H23 | 119.6 |
| C3—C4—C5 | 119.0 (3) | C24—C23—H23 | 119.6 |
| C3—C4—H4 | 120.5 | C29—C24—C25 | 116.7 (3) |
| C5—C4—H4 | 120.5 | C29—C24—C23 | 115.8 (3) |
| C4—C5—C6 | 120.1 (3) | C25—C24—C23 | 127.5 (3) |
| C4—C5—H5 | 120.0 | C26—C25—C24 | 120.5 (3) |
| C6—C5—H5 | 120.0 | C26—C25—H25 | 119.7 |
| C1—C6—C5 | 119.3 (2) | C24—C25—H25 | 119.7 |
| C1—C6—C7 | 121.1 (2) | C25—C26—C27 | 122.3 (3) |
| C5—C6—C7 | 119.6 (2) | C25—C26—H26 | 118.9 |
| O2—C7—C8 | 123.3 (2) | C27—C26—H26 | 118.9 |
| O2—C7—C6 | 112.9 (2) | C28—C27—C26 | 118.8 (3) |
| C8—C7—C6 | 123.7 (2) | C28—C27—H27 | 120.6 |
| C7—C8—C17 | 126.3 (2) | C26—C27—H27 | 120.6 |
| C7—C8—C9 | 126.3 (2) | C27—C28—C29 | 119.0 (2) |

| | | | |
|--------------|------------|-----------------|------------|
| C17—C8—C9 | 107.4 (2) | C27—C28—C18 | 136.0 (2) |
| C19—C9—C10 | 127.0 (2) | C29—C28—C18 | 104.9 (2) |
| C19—C9—C8 | 106.3 (2) | C24—C29—C28 | 122.7 (3) |
| C10—C9—C8 | 126.7 (2) | C24—C29—C20 | 124.3 (3) |
| C19—C9—H9 | 90.7 | C28—C29—C20 | 112.9 (2) |
| C10—C9—H9 | 90.7 | C31—C30—C39 | 118.9 (2) |
| C8—C9—H9 | 90.7 | C31—C30—C17 | 119.2 (2) |
| O1—C10—C9 | 124.1 (2) | C39—C30—C17 | 121.7 (2) |
| O1—C10—C11 | 114.9 (2) | C30—C31—C32 | 121.6 (3) |
| C9—C10—C11 | 121.0 (2) | C30—C31—H31 | 119.2 |
| C16—C11—C12 | 119.5 (2) | C32—C31—H31 | 119.2 |
| C16—C11—C10 | 121.0 (2) | C33—C32—C31 | 119.8 (3) |
| C12—C11—C10 | 119.4 (2) | C33—C32—H32 | 120.1 |
| C13—C12—C11 | 120.5 (3) | C31—C32—H32 | 120.1 |
| C13—C12—H12 | 119.7 | C32—C33—C34 | 120.9 (3) |
| C11—C12—H12 | 119.7 | C32—C33—H33 | 119.6 |
| C14—C13—C12 | 118.8 (3) | C34—C33—H33 | 119.6 |
| C14—C13—H13 | 120.6 | C33—C34—C35 | 121.6 (3) |
| C12—C13—H13 | 120.6 | C33—C34—C39 | 119.4 (3) |
| C13—C14—C15 | 121.9 (3) | C35—C34—C39 | 119.0 (3) |
| C13—C14—C11 | 118.6 (2) | C36—C35—C34 | 121.6 (3) |
| C15—C14—C11 | 119.5 (2) | C36—C35—H35 | 119.2 |
| C14—C15—C16 | 118.8 (3) | C34—C35—H35 | 119.2 |
| C14—C15—H15 | 120.6 | C35—C36—C37 | 119.8 (3) |
| C16—C15—H15 | 120.6 | C35—C36—H36 | 120.1 |
| C11—C16—C15 | 120.5 (3) | C37—C36—H36 | 120.1 |
| C11—C16—H16 | 119.7 | C38—C37—C36 | 120.6 (3) |
| C15—C16—H16 | 119.7 | C38—C37—H37 | 119.7 |
| C18—C17—C8 | 107.4 (2) | C36—C37—H37 | 119.7 |
| C18—C17—C30 | 124.2 (2) | C37—C38—C39 | 120.7 (2) |
| C8—C17—C30 | 128.3 (2) | C37—C38—C40 | 117.1 (2) |
| C17—C18—C19 | 110.0 (2) | C39—C38—C40 | 121.0 (2) |
| C17—C18—C28 | 139.7 (2) | C34—C39—C38 | 117.6 (2) |
| C19—C18—C28 | 109.9 (2) | C34—C39—C30 | 118.2 (2) |
| C9—C19—C18 | 108.9 (2) | C38—C39—C30 | 124.2 (2) |
| C9—C19—C20 | 143.3 (2) | O3—C40—O4 | 124.3 (2) |
| C18—C19—C20 | 107.2 (2) | O3—C40—C38 | 120.0 (2) |
| C21—C20—C29 | 117.4 (2) | O4—C40—C38 | 115.5 (2) |
| C21—C20—C19 | 137.5 (3) | C40—O4—H4' | 104 (3) |
| | | | |
| C6—C1—C2—C3 | -1.0 (4) | C29—C20—C21—C22 | 1.8 (4) |
| C1—C2—C3—C4 | 2.5 (5) | C19—C20—C21—C22 | 178.7 (3) |
| C1—C2—C3—C12 | -177.0 (2) | C20—C21—C22—C23 | -2.0 (5) |
| C2—C3—C4—C5 | -1.1 (5) | C21—C22—C23—C24 | 0.0 (5) |
| C12—C3—C4—C5 | 178.4 (2) | C22—C23—C24—C29 | 2.0 (5) |
| C3—C4—C5—C6 | -1.8 (4) | C22—C23—C24—C25 | -175.2 (3) |
| C2—C1—C6—C5 | -1.8 (4) | C29—C24—C25—C26 | 0.1 (5) |
| C2—C1—C6—C7 | -179.7 (2) | C23—C24—C25—C26 | 177.3 (3) |

| | | | |
|-----------------|------------|-----------------|------------|
| C4—C5—C6—C1 | 3.2 (4) | C24—C25—C26—C27 | 0.8 (5) |
| C4—C5—C6—C7 | -178.8 (2) | C25—C26—C27—C28 | -0.5 (5) |
| C1—C6—C7—O2 | 133.8 (3) | C26—C27—C28—C29 | -0.6 (4) |
| C5—C6—C7—O2 | -44.1 (3) | C26—C27—C28—C18 | -176.6 (3) |
| C1—C6—C7—C8 | -46.5 (4) | C17—C18—C28—C27 | 3.0 (6) |
| C5—C6—C7—C8 | 135.6 (3) | C19—C18—C28—C27 | 174.3 (3) |
| O2—C7—C8—C17 | 161.1 (3) | C17—C18—C28—C29 | -173.4 (3) |
| C6—C7—C8—C17 | -18.6 (4) | C19—C18—C28—C29 | -2.2 (3) |
| O2—C7—C8—C9 | -19.6 (4) | C25—C24—C29—C28 | -1.2 (4) |
| C6—C7—C8—C9 | 160.8 (2) | C23—C24—C29—C28 | -178.8 (3) |
| C7—C8—C9—C19 | -177.7 (2) | C25—C24—C29—C20 | 175.4 (3) |
| C17—C8—C9—C19 | 1.8 (3) | C23—C24—C29—C20 | -2.2 (4) |
| C7—C8—C9—C10 | 4.5 (4) | C27—C28—C29—C24 | 1.5 (4) |
| C17—C8—C9—C10 | -176.0 (3) | C18—C28—C29—C24 | 178.7 (2) |
| C19—C9—C10—O1 | -155.4 (3) | C27—C28—C29—C20 | -175.5 (2) |
| C8—C9—C10—O1 | 21.9 (5) | C18—C28—C29—C20 | 1.7 (3) |
| C19—C9—C10—C11 | 24.2 (4) | C21—C20—C29—C24 | 0.3 (4) |
| C8—C9—C10—C11 | -158.5 (2) | C19—C20—C29—C24 | -177.5 (2) |
| O1—C10—C11—C16 | -131.2 (3) | C21—C20—C29—C28 | 177.2 (2) |
| C9—C10—C11—C16 | 49.2 (4) | C19—C20—C29—C28 | -0.6 (3) |
| O1—C10—C11—C12 | 44.0 (4) | C18—C17—C30—C31 | -61.0 (4) |
| C9—C10—C11—C12 | -135.7 (3) | C8—C17—C30—C31 | 122.7 (3) |
| C16—C11—C12—C13 | -2.0 (4) | C18—C17—C30—C39 | 123.9 (3) |
| C10—C11—C12—C13 | -177.2 (3) | C8—C17—C30—C39 | -52.3 (4) |
| C11—C12—C13—C14 | 2.2 (5) | C39—C30—C31—C32 | 7.0 (4) |
| C12—C13—C14—C15 | -0.7 (5) | C17—C30—C31—C32 | -168.2 (3) |
| C12—C13—C14—C11 | 177.5 (2) | C30—C31—C32—C33 | 2.4 (5) |
| C13—C14—C15—C16 | -0.9 (5) | C31—C32—C33—C34 | -6.1 (5) |
| C11—C14—C15—C16 | -179.1 (2) | C32—C33—C34—C35 | -178.8 (3) |
| C12—C11—C16—C15 | 0.3 (4) | C32—C33—C34—C39 | 0.3 (4) |
| C10—C11—C16—C15 | 175.5 (3) | C33—C34—C35—C36 | 178.6 (3) |
| C14—C15—C16—C11 | 1.1 (4) | C39—C34—C35—C36 | -0.6 (4) |
| C7—C8—C17—C18 | 178.7 (2) | C34—C35—C36—C37 | -4.6 (5) |
| C9—C8—C17—C18 | -0.7 (3) | C35—C36—C37—C38 | 2.8 (5) |
| C7—C8—C17—C30 | -4.5 (4) | C36—C37—C38—C39 | 4.2 (4) |
| C9—C8—C17—C30 | 176.1 (2) | C36—C37—C38—C40 | -163.6 (3) |
| C8—C17—C18—C19 | -0.6 (3) | C33—C34—C39—C38 | -171.9 (2) |
| C30—C17—C18—C19 | -177.5 (2) | C35—C34—C39—C38 | 7.2 (4) |
| C8—C17—C18—C28 | 170.7 (3) | C33—C34—C39—C30 | 8.9 (4) |
| C30—C17—C18—C28 | -6.3 (5) | C35—C34—C39—C30 | -171.9 (2) |
| C10—C9—C19—C18 | 175.7 (3) | C37—C38—C39—C34 | -9.1 (4) |
| C8—C9—C19—C18 | -2.1 (3) | C40—C38—C39—C34 | 158.2 (2) |
| C10—C9—C19—C20 | 5.2 (6) | C37—C38—C39—C30 | 170.0 (3) |
| C8—C9—C19—C20 | -172.6 (3) | C40—C38—C39—C30 | -22.7 (4) |
| C17—C18—C19—C9 | 1.8 (3) | C31—C30—C39—C34 | -12.4 (4) |
| C28—C18—C19—C9 | -172.2 (2) | C17—C30—C39—C34 | 162.6 (2) |
| C17—C18—C19—C20 | 175.8 (2) | C31—C30—C39—C38 | 168.5 (2) |
| C28—C18—C19—C20 | 1.8 (3) | C17—C30—C39—C38 | -16.5 (4) |

| | | | |
|-----------------|------------|----------------|-----------|
| C9—C19—C20—C21 | -7.3 (6) | C37—C38—C40—O3 | 125.5 (3) |
| C18—C19—C20—C21 | -177.9 (3) | C39—C38—C40—O3 | -42.2 (4) |
| C9—C19—C20—C29 | 169.8 (3) | C37—C38—C40—O4 | -50.1 (3) |
| C18—C19—C20—C29 | -0.7 (3) | C39—C38—C40—O4 | 142.2 (3) |

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C18—C20/C28/C29 ring, Cg2 is the centroid of the C24—C29 ring and Cg3 is the centroid of the C11—C16 ring.

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|------------------------------|-------------|---------------|-----------------------|-------------------------|
| O4—H4'...O3 ⁱ | 0.84 (1) | 1.81 (1) | 2.649 (3) | 177 (5) |
| C26—H26)...O3 ⁱⁱ | 0.93 | 2.52 | 3.416 (4) | 163 |
| C32—H32)...O2 ⁱⁱⁱ | 0.93 | 2.47 | 3.301 (4) | 149 |
| C35—H35)...C12 ^{iv} | 0.93 | 2.74 | 3.619 (3) | 157 |
| C2—H2...Cg1 ^v | 0.93 | 2.87 | 3.577 (3) | 134 |
| C12—H12...Cg2 ^{vi} | 0.93 | 2.84 | 3.725 (3) | 160 |
| C21—H21...Cg3 | 0.93 | 2.57 | 3.425 (3) | 152 |

Symmetry codes: (i) $-x, -y+1, -z+2$; (ii) $-x+1, -y+1, -z+2$; (iii) $x+1, y, z$; (iv) $-x+1, -y, -z+3$; (v) $-x+1, -y, -z+2$; (vi) $x-1, y, z$.