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# Rubrene endoperoxide acetone monosolvate

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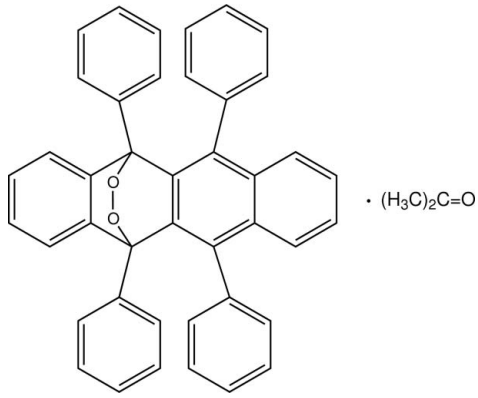
Key indicators: single-crystal X-ray study; $T$ = 90 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.049; $wR$ factor = 0.119; data-to-parameter ratio = 17.6.

The title acetone solvate, $C_{42}H_{28}O_2\cdot C_3H_6O$ [systematic name: 1,3,10,12-tetraphenyl-19,20-dioxapentacyclo[10.6.2.0$^{2,11}$.0$^{4,9}$.0$^{13,18}$]icosa-2(11),3,5,7,9,13,15,17-octaene acetone monosolvate], is a photooxygenation product of rubrene (systematic name: 5,6,11,12-tetraphenyltetracene). The molecule bends at the bridgehead atoms, which are linked by the O—O transannular bond, with a dihedral angle of 49.21 (6)° between the benzene ring and the naphthalene ring system of the tetracene unit. In the crystal, the rubrene molecules are linked by C—H···O hydrogen bonds into a column along the $c$ axis. The acetone solvent molecules form a dimer around a crystallographic inversion centre through a carbonyl–carbonyl dipolar interaction. A C—H···O hydrogen bond between the rubrene and acetone molecules is also observed.

## Related literature

For related structures, see: Brown & Ehrenberg (1984); Izuoka *et al.* (1997); Schuster *et al.* (2002); Usman *et al.* (2003); Wang (2008). For background to photooxygenation of polycyclic aromatic hydrocarbons, see: Sakai *et al.* (1995).




## Experimental

### Crystal data

$C_{42}H_{28}O_2\cdot C_3H_6O$
$M_r$ = 622.72
Monoclinic, $P2_1/c$
$a$ = 11.1592 (6) Å
$b$ = 21.2248 (11) Å
$c$ = 13.6121 (7) Å
$\beta$ = 103.113 (1)°
$V$ = 3140.0 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 90 K
0.15 × 0.07 × 0.05 mm

### Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.988, $T_{max}$ = 0.996
20867 measured reflections
7642 independent reflections
5257 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.046

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.119
$S$ = 1.03
7642 reflections
435 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.29 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C28—H28···O3$^{i}$ | 0.95 | 2.53 | 3.437 (2) | 159 |
| C35—H35···O1$^{ii}$ | 0.95 | 2.59 | 3.408 (2) | 144 |

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* and *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5077).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o995–o996 [doi:10.1107/S1600536812008835]

## Rubrene endoperoxide acetone monosolvate

**Kiyoaki Shinashi and Akira Uchida**

### Comment

Polycyclic aromatic hydrocarbons (PAHs) in solution are known to react with molecular oxygen to form endoperoxides when PAHs are irradiated by light with their absorption wavelength. The photooxygenation of PAHs occur due to 1,4-cycloaddition of singlet oxygen to the ground state of a PAH molecule (Sakai *et al.*, 1995). We present here the crystal structure of the title compound.

The molecule bends at the bridgehead atoms, C9 and C10, with a dihedral angle of 49.21 (6)° between the benzene C1–C4/C14/C13 ring and the naphthalene C5–C8/C17/C11/C15/C16/C12/C18 ring system (Figure 1). The adjacent phenyl groups [C19—C24 (centroid *Cg*1), C25—C30 (centroid *Cg*2), C31—C36 (centroid *Cg*3), C37—C42 (centroid *Cg*4)] on either side of the molecule adopt a splayed face-to-face geometry with *Cg*1···*Cg*3 distance of 3.455 (1) Å and *Cg*2···*Cg*4 distance of 3.491 (1) Å (Figure 2). The molecules related by glide plane are linked by C—H···O hydrogen bonds along the *c* axis.

### Experimental

Rubrene was purchased from Sigma-Aldrich. An acetone solution of the compound was exposed to sunlight until the solution became colourless. Crystals of the title compound were obtained from the solution placed in the dark.

### Refinement

All H atoms were placed in geometrical positions and constrained to ride on their parent atoms with C—H = 0.95 for aromatic H atoms and 0.98 Å for $CH_3$ type H atoms, respectively. $U_{iso}$(H) values were set at 1.2 times $U_{eq}$(C) for aromatic H atoms and 1.5 times for methyl H atoms.

### Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

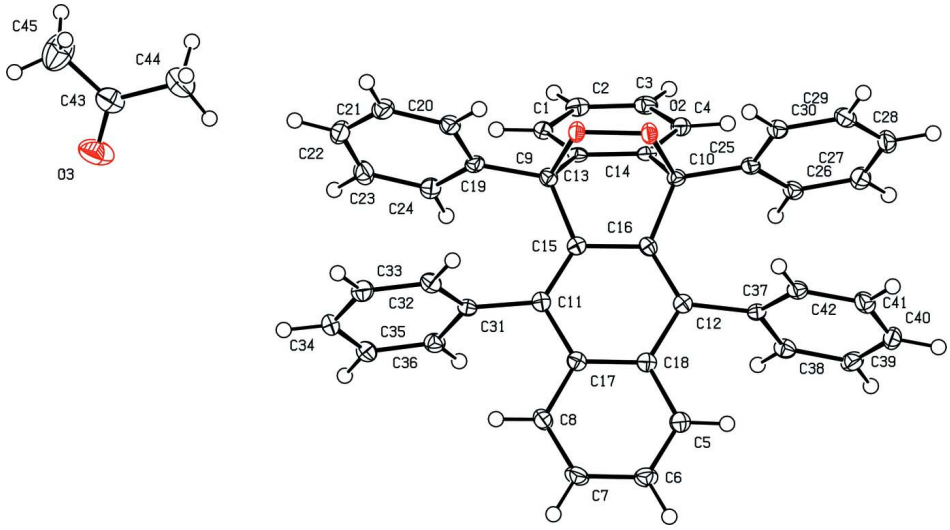


**Figure 1**

The molecular structure of the title compound, showing displacement ellipsoids at the 50% probability level.

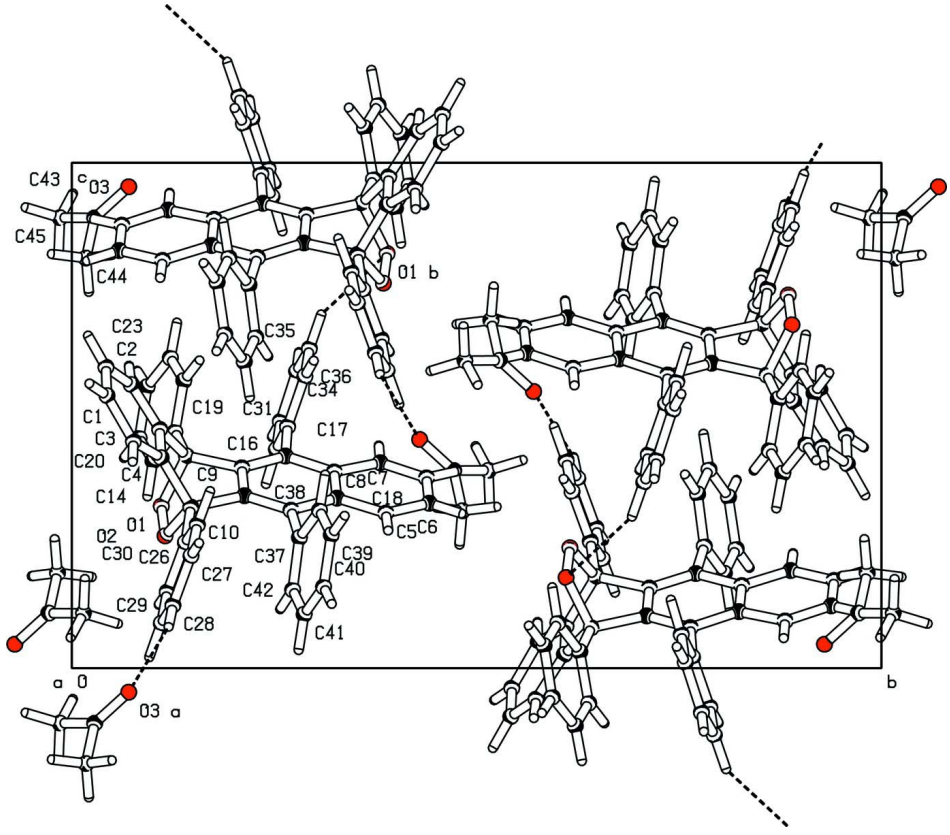


**Figure 2**

The crystal packing of the title compound viewed along the *a* axis. The dashed lines indicate C—H···O intermolecular interactions.

**1,3,10,12-tetraphenyl-19,20- dioxapentacyclo[10.6.2.0$^{2,11}$.0$^{4,9}$.0$^{13,18}$]icosa- 2(11),3,5,7,9,13,15,17-octaene acetone monosolvate**

*Crystal data*

$C_{42}H_{28}O_2 \cdot C_3H_6O$
$M_r$ = 622.72
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 11.1592 (6) Å
$b$ = 21.2248 (11) Å
$c$ = 13.6121 (7) Å
$\beta$ = 103.113 (1)°
$V$ = 3140.0 (3) Å$^3$
$Z$ = 4
$F(000)$ = 1312
$D_x$ = 1.317 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3033 reflections
$\theta$ = 2.3–26.7°
$\mu$ = 0.08 mm$^{-1}$
$T$ = 90 K
Plate, colourless
0.15 × 0.07 × 0.05 mm

*Data collection*

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.366 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.988, $T_{max}$ = 0.996
20867 measured reflections
7642 independent reflections
5257 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.046
$\theta_{max}$ = 28.1°, $\theta_{min}$ = 1.8°
$h$ = −14→11
$k$ = −28→23
$l$ = −17→18

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.119
$S$ = 1.03
7642 reflections
435 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
$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.4529P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.29 e Å$^{-3}$

*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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.18118 (10) | 0.61054 (5) | 0.17937 (8) | 0.0153 (2) |
| O2 | 0.08624 (10) | 0.61524 (5) | 0.23817 (8) | 0.0159 (2) |
| O3 | 0.56171 (15) | 0.92967 (6) | 0.04751 (10) | 0.0425 (4) |

| | | | | |
|---|---|---|---|---|
| C1 | 0.00522 (15) | 0.57338 (7) | −0.06128 (11) | 0.0157 (3) |
| H1 | 0.0677 | 0.5707 | −0.0983 | 0.019* |
| C2 | −0.10563 (15) | 0.54225 (7) | −0.09559 (12) | 0.0172 (3) |
| H2 | −0.1206 | 0.5200 | −0.1578 | 0.021* |
| C3 | −0.19435 (15) | 0.54349 (7) | −0.03941 (12) | 0.0179 (3) |
| H3 | −0.2699 | 0.5219 | −0.0630 | 0.021* |
| C4 | −0.17376 (15) | 0.57614 (7) | 0.05126 (12) | 0.0161 (3) |
| H4 | −0.2333 | 0.5752 | 0.0912 | 0.019* |
| C5 | 0.00268 (15) | 0.88782 (7) | 0.19322 (12) | 0.0174 (3) |
| H5 | −0.0624 | 0.8908 | 0.2274 | 0.021* |
| C6 | 0.06089 (16) | 0.94148 (7) | 0.17353 (12) | 0.0201 (4) |
| H6 | 0.0358 | 0.9812 | 0.1939 | 0.024* |
| C7 | 0.15725 (16) | 0.93803 (7) | 0.12347 (12) | 0.0219 (4) |
| H7 | 0.1977 | 0.9754 | 0.1103 | 0.026* |
| C8 | 0.19347 (15) | 0.88095 (7) | 0.09338 (12) | 0.0187 (3) |
| H8 | 0.2592 | 0.8792 | 0.0597 | 0.022* |
| C9 | 0.14375 (14) | 0.64156 (7) | 0.07997 (11) | 0.0139 (3) |
| C10 | −0.02556 (14) | 0.64712 (7) | 0.18133 (11) | 0.0132 (3) |
| C11 | 0.17197 (14) | 0.76393 (7) | 0.08028 (11) | 0.0141 (3) |
| C12 | −0.02111 (14) | 0.77070 (7) | 0.18730 (11) | 0.0135 (3) |
| C13 | 0.02509 (14) | 0.60853 (7) | 0.02736 (11) | 0.0128 (3) |
| C14 | −0.06515 (14) | 0.61037 (6) | 0.08337 (11) | 0.0135 (3) |
| C15 | 0.11456 (14) | 0.70993 (7) | 0.10355 (11) | 0.0131 (3) |
| C16 | 0.01819 (14) | 0.71332 (7) | 0.15861 (11) | 0.0131 (3) |
| C17 | 0.13408 (14) | 0.82406 (7) | 0.11186 (11) | 0.0146 (3) |
| C18 | 0.03779 (14) | 0.82748 (7) | 0.16346 (11) | 0.0145 (3) |
| C19 | 0.25208 (14) | 0.62739 (7) | 0.03296 (12) | 0.0146 (3) |
| C20 | 0.35961 (15) | 0.60032 (7) | 0.08973 (13) | 0.0179 (3) |
| H20 | 0.3679 | 0.5935 | 0.1599 | 0.021* |
| C21 | 0.45484 (16) | 0.58325 (7) | 0.04452 (14) | 0.0230 (4) |
| H21 | 0.5275 | 0.5646 | 0.0839 | 0.028* |
| C22 | 0.44413 (16) | 0.59327 (8) | −0.05728 (14) | 0.0254 (4) |
| H22 | 0.5084 | 0.5807 | −0.0884 | 0.030* |
| C23 | 0.33927 (16) | 0.62178 (8) | −0.11395 (13) | 0.0226 (4) |
| H23 | 0.3327 | 0.6299 | −0.1836 | 0.027* |
| C24 | 0.24402 (15) | 0.63844 (7) | −0.06923 (12) | 0.0176 (3) |
| H24 | 0.1722 | 0.6577 | −0.1088 | 0.021* |
| C25 | −0.11396 (15) | 0.64040 (7) | 0.25125 (11) | 0.0150 (3) |
| C26 | −0.23815 (15) | 0.65513 (7) | 0.21634 (12) | 0.0163 (3) |
| H26 | −0.2665 | 0.6708 | 0.1497 | 0.020* |
| C27 | −0.32065 (15) | 0.64729 (7) | 0.27750 (12) | 0.0204 (4) |
| H27 | −0.4051 | 0.6572 | 0.2525 | 0.024* |
| C28 | −0.28005 (16) | 0.62495 (7) | 0.37559 (13) | 0.0220 (4) |
| H28 | −0.3364 | 0.6196 | 0.4178 | 0.026* |
| C29 | −0.15715 (16) | 0.61064 (7) | 0.41095 (13) | 0.0208 (4) |
| H29 | −0.1289 | 0.5957 | 0.4780 | 0.025* |
| C30 | −0.07414 (15) | 0.61793 (7) | 0.34927 (12) | 0.0173 (3) |
| H30 | 0.0101 | 0.6075 | 0.3742 | 0.021* |
| C31 | 0.27087 (14) | 0.76655 (7) | 0.02205 (12) | 0.0147 (3) |

| | | | | |
|---|---|---|---|---|
| C32 | 0.39281 (14) | 0.75275 (7) | 0.06683 (12) | 0.0167 (3) |
| H32 | 0.4146 | 0.7399 | 0.1354 | 0.020* |
| C33 | 0.48295 (15) | 0.75768 (7) | 0.01173 (13) | 0.0208 (4) |
| H33 | 0.5658 | 0.7473 | 0.0424 | 0.025* |
| C34 | 0.45275 (16) | 0.77769 (7) | −0.08750 (13) | 0.0218 (4) |
| H34 | 0.5146 | 0.7806 | −0.1251 | 0.026* |
| C35 | 0.33226 (16) | 0.79350 (7) | −0.13213 (13) | 0.0207 (4) |
| H35 | 0.3116 | 0.8081 | −0.1999 | 0.025* |
| C36 | 0.24205 (15) | 0.78793 (7) | −0.07754 (12) | 0.0174 (3) |
| H36 | 0.1595 | 0.7988 | −0.1083 | 0.021* |
| C37 | −0.12262 (14) | 0.78122 (7) | 0.24124 (11) | 0.0144 (3) |
| C38 | −0.23453 (15) | 0.80595 (7) | 0.18777 (12) | 0.0178 (3) |
| H38 | −0.2470 | 0.8129 | 0.1172 | 0.021* |
| C39 | −0.32743 (15) | 0.82053 (7) | 0.23616 (13) | 0.0217 (4) |
| H39 | −0.4033 | 0.8370 | 0.1988 | 0.026* |
| C40 | −0.30963 (16) | 0.81102 (7) | 0.33911 (14) | 0.0224 (4) |
| H40 | −0.3733 | 0.8210 | 0.3725 | 0.027* |
| C41 | −0.19894 (16) | 0.78702 (7) | 0.39326 (13) | 0.0205 (4) |
| H41 | −0.1871 | 0.7802 | 0.4638 | 0.025* |
| C42 | −0.10512 (15) | 0.77285 (7) | 0.34508 (12) | 0.0159 (3) |
| H42 | −0.0287 | 0.7574 | 0.3831 | 0.019* |
| C43 | 0.59016 (18) | 0.97168 (8) | 0.10896 (13) | 0.0279 (4) |
| C44 | 0.5198 (2) | 0.98239 (9) | 0.18860 (15) | 0.0382 (5) |
| H44A | 0.4545 | 0.9507 | 0.1822 | 0.057* |
| H44B | 0.4831 | 1.0246 | 0.1806 | 0.057* |
| H44C | 0.5755 | 0.9789 | 0.2553 | 0.057* |
| C45 | 0.6960 (2) | 1.01456 (11) | 0.10861 (17) | 0.0483 (6) |
| H45A | 0.7428 | 0.9987 | 0.0610 | 0.072* |
| H45B | 0.7495 | 1.0162 | 0.1764 | 0.072* |
| H45C | 0.6651 | 1.0569 | 0.0882 | 0.072* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0139 (6) | 0.0193 (6) | 0.0138 (5) | 0.0032 (4) | 0.0054 (5) | 0.0022 (4) |
| O2 | 0.0144 (6) | 0.0206 (6) | 0.0140 (5) | 0.0026 (4) | 0.0060 (5) | 0.0033 (4) |
| O3 | 0.0684 (11) | 0.0293 (7) | 0.0296 (8) | 0.0031 (7) | 0.0111 (8) | −0.0067 (6) |
| C1 | 0.0177 (8) | 0.0144 (7) | 0.0154 (8) | 0.0020 (6) | 0.0049 (7) | 0.0025 (6) |
| C2 | 0.0223 (9) | 0.0141 (7) | 0.0136 (8) | 0.0013 (6) | 0.0004 (7) | −0.0003 (6) |
| C3 | 0.0161 (8) | 0.0135 (7) | 0.0223 (8) | −0.0017 (6) | 0.0006 (7) | 0.0006 (6) |
| C4 | 0.0152 (8) | 0.0138 (7) | 0.0194 (8) | 0.0001 (6) | 0.0042 (7) | 0.0005 (6) |
| C5 | 0.0206 (8) | 0.0174 (8) | 0.0141 (8) | 0.0005 (6) | 0.0039 (7) | −0.0003 (6) |
| C6 | 0.0268 (9) | 0.0138 (8) | 0.0197 (8) | −0.0006 (7) | 0.0054 (7) | −0.0019 (6) |
| C7 | 0.0292 (10) | 0.0134 (8) | 0.0248 (9) | −0.0050 (7) | 0.0093 (8) | 0.0004 (6) |
| C8 | 0.0217 (9) | 0.0179 (8) | 0.0184 (8) | −0.0028 (7) | 0.0083 (7) | 0.0009 (6) |
| C9 | 0.0150 (8) | 0.0149 (7) | 0.0120 (7) | 0.0001 (6) | 0.0031 (6) | 0.0007 (6) |
| C10 | 0.0120 (7) | 0.0120 (7) | 0.0150 (8) | 0.0007 (6) | 0.0021 (6) | 0.0009 (6) |
| C11 | 0.0130 (8) | 0.0167 (7) | 0.0120 (7) | 0.0001 (6) | 0.0014 (6) | 0.0006 (6) |
| C12 | 0.0137 (8) | 0.0154 (7) | 0.0103 (7) | 0.0004 (6) | 0.0004 (6) | 0.0001 (6) |
| C13 | 0.0135 (8) | 0.0100 (7) | 0.0139 (8) | 0.0018 (6) | 0.0009 (6) | 0.0029 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C14 | 0.0164 (8) | 0.0088 (7) | 0.0149 (8) | 0.0017 (6) | 0.0029 (6) | 0.0014 (6) |
| C15 | 0.0130 (7) | 0.0140 (7) | 0.0111 (7) | 0.0006 (6) | 0.0004 (6) | −0.0002 (6) |
| C16 | 0.0130 (8) | 0.0153 (7) | 0.0106 (7) | −0.0013 (6) | 0.0017 (6) | 0.0013 (6) |
| C17 | 0.0166 (8) | 0.0151 (7) | 0.0115 (7) | −0.0011 (6) | 0.0022 (6) | −0.0003 (6) |
| C18 | 0.0163 (8) | 0.0151 (7) | 0.0112 (7) | −0.0003 (6) | 0.0015 (6) | −0.0005 (6) |
| C19 | 0.0154 (8) | 0.0103 (7) | 0.0184 (8) | −0.0014 (6) | 0.0041 (7) | −0.0033 (6) |
| C20 | 0.0169 (8) | 0.0150 (8) | 0.0220 (9) | −0.0016 (6) | 0.0049 (7) | −0.0002 (6) |
| C21 | 0.0176 (9) | 0.0169 (8) | 0.0356 (10) | 0.0006 (7) | 0.0085 (8) | 0.0003 (7) |
| C22 | 0.0228 (9) | 0.0191 (8) | 0.0389 (11) | −0.0024 (7) | 0.0167 (9) | −0.0066 (7) |
| C23 | 0.0259 (10) | 0.0213 (8) | 0.0235 (9) | −0.0069 (7) | 0.0117 (8) | −0.0048 (7) |
| C24 | 0.0177 (8) | 0.0163 (8) | 0.0189 (8) | −0.0026 (6) | 0.0044 (7) | −0.0037 (6) |
| C25 | 0.0191 (8) | 0.0100 (7) | 0.0172 (8) | −0.0025 (6) | 0.0067 (7) | −0.0024 (6) |
| C26 | 0.0190 (8) | 0.0130 (7) | 0.0176 (8) | −0.0034 (6) | 0.0054 (7) | −0.0013 (6) |
| C27 | 0.0180 (9) | 0.0184 (8) | 0.0261 (9) | −0.0034 (7) | 0.0076 (7) | −0.0050 (7) |
| C28 | 0.0276 (10) | 0.0185 (8) | 0.0244 (9) | −0.0049 (7) | 0.0155 (8) | −0.0045 (7) |
| C29 | 0.0302 (10) | 0.0152 (8) | 0.0191 (9) | −0.0031 (7) | 0.0101 (8) | 0.0001 (6) |
| C30 | 0.0200 (9) | 0.0140 (7) | 0.0186 (8) | −0.0008 (6) | 0.0058 (7) | 0.0000 (6) |
| C31 | 0.0163 (8) | 0.0107 (7) | 0.0178 (8) | −0.0022 (6) | 0.0051 (7) | −0.0012 (6) |
| C32 | 0.0172 (8) | 0.0164 (8) | 0.0157 (8) | −0.0027 (6) | 0.0024 (7) | −0.0003 (6) |
| C33 | 0.0139 (8) | 0.0184 (8) | 0.0297 (10) | −0.0003 (6) | 0.0041 (7) | 0.0003 (7) |
| C34 | 0.0225 (9) | 0.0187 (8) | 0.0286 (10) | 0.0000 (7) | 0.0150 (8) | 0.0030 (7) |
| C35 | 0.0262 (9) | 0.0192 (8) | 0.0189 (8) | 0.0023 (7) | 0.0095 (7) | 0.0042 (6) |
| C36 | 0.0168 (8) | 0.0165 (8) | 0.0185 (8) | 0.0008 (6) | 0.0029 (7) | 0.0020 (6) |
| C37 | 0.0167 (8) | 0.0100 (7) | 0.0175 (8) | −0.0012 (6) | 0.0057 (7) | −0.0020 (6) |
| C38 | 0.0176 (8) | 0.0159 (8) | 0.0191 (8) | −0.0018 (6) | 0.0026 (7) | 0.0012 (6) |
| C39 | 0.0154 (8) | 0.0149 (8) | 0.0353 (10) | 0.0012 (6) | 0.0067 (8) | −0.0004 (7) |
| C40 | 0.0219 (9) | 0.0171 (8) | 0.0327 (10) | −0.0037 (7) | 0.0155 (8) | −0.0068 (7) |
| C41 | 0.0271 (9) | 0.0167 (8) | 0.0200 (9) | −0.0066 (7) | 0.0102 (7) | −0.0040 (6) |
| C42 | 0.0177 (8) | 0.0124 (7) | 0.0175 (8) | −0.0029 (6) | 0.0037 (7) | −0.0023 (6) |
| C43 | 0.0367 (11) | 0.0251 (9) | 0.0203 (9) | 0.0068 (8) | 0.0032 (8) | 0.0025 (7) |
| C44 | 0.0422 (13) | 0.0383 (11) | 0.0361 (11) | 0.0029 (9) | 0.0129 (10) | −0.0065 (9) |
| C45 | 0.0463 (14) | 0.0614 (15) | 0.0359 (12) | −0.0137 (11) | 0.0068 (11) | 0.0054 (11) |

*Geometric parameters (Å, º)*

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| O1—O2 | 1.4689 (14) | C23—C24 | 1.385 (2) |
| O1—C9 | 1.4772 (18) | C23—H23 | 0.9500 |
| O2—C10 | 1.4749 (18) | C24—H24 | 0.9500 |
| O3—C43 | 1.214 (2) | C25—C30 | 1.391 (2) |
| C1—C2 | 1.387 (2) | C25—C26 | 1.395 (2) |
| C1—C13 | 1.393 (2) | C26—C27 | 1.384 (2) |
| C1—H1 | 0.9500 | C26—H26 | 0.9500 |
| C2—C3 | 1.382 (2) | C27—C28 | 1.392 (2) |
| C2—H2 | 0.9500 | C27—H27 | 0.9500 |
| C3—C4 | 1.388 (2) | C28—C29 | 1.380 (2) |
| C3—H3 | 0.9500 | C28—H28 | 0.9500 |
| C4—C14 | 1.396 (2) | C29—C30 | 1.393 (2) |
| C4—H4 | 0.9500 | C29—H29 | 0.9500 |
| C5—C6 | 1.367 (2) | C30—H30 | 0.9500 |
| C5—C18 | 1.425 (2) | C31—C32 | 1.390 (2) |

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| C5—H5 | 0.9500 | C31—C36 | 1.396 (2) |
| C6—C7 | 1.399 (2) | C32—C33 | 1.388 (2) |
| C6—H6 | 0.9500 | C32—H32 | 0.9500 |
| C7—C8 | 1.369 (2) | C33—C34 | 1.383 (2) |
| C7—H7 | 0.9500 | C33—H33 | 0.9500 |
| C8—C17 | 1.427 (2) | C34—C35 | 1.385 (2) |
| C8—H8 | 0.9500 | C34—H34 | 0.9500 |
| C9—C19 | 1.521 (2) | C35—C36 | 1.385 (2) |
| C9—C13 | 1.526 (2) | C35—H35 | 0.9500 |
| C9—C15 | 1.537 (2) | C36—H36 | 0.9500 |
| C10—C14 | 1.521 (2) | C37—C42 | 1.394 (2) |
| C10—C25 | 1.525 (2) | C37—C38 | 1.397 (2) |
| C10—C16 | 1.542 (2) | C38—C39 | 1.384 (2) |
| C11—C15 | 1.384 (2) | C38—H38 | 0.9500 |
| C11—C17 | 1.440 (2) | C39—C40 | 1.385 (2) |
| C11—C31 | 1.499 (2) | C39—H39 | 0.9500 |
| C12—C16 | 1.380 (2) | C40—C41 | 1.384 (2) |
| C12—C18 | 1.444 (2) | C40—H40 | 0.9500 |
| C12—C37 | 1.500 (2) | C41—C42 | 1.389 (2) |
| C13—C14 | 1.395 (2) | C41—H41 | 0.9500 |
| C15—C16 | 1.445 (2) | C42—H42 | 0.9500 |
| C17—C18 | 1.413 (2) | C43—C45 | 1.492 (3) |
| C19—C24 | 1.393 (2) | C43—C44 | 1.493 (2) |
| C19—C20 | 1.395 (2) | C44—H44A | 0.9800 |
| C20—C21 | 1.391 (2) | C44—H44B | 0.9800 |
| C20—H20 | 0.9500 | C44—H44C | 0.9800 |
| C21—C22 | 1.380 (2) | C45—H45A | 0.9800 |
| C21—H21 | 0.9500 | C45—H45B | 0.9800 |
| C22—C23 | 1.386 (2) | C45—H45C | 0.9800 |
| C22—H22 | 0.9500 | | |

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| O2—O1—C9 | 112.32 (10) | C24—C23—H23 | 119.9 |
| O1—O2—C10 | 111.97 (9) | C22—C23—H23 | 119.9 |
| C2—C1—C13 | 120.10 (14) | C23—C24—C19 | 120.90 (16) |
| C2—C1—H1 | 120.0 | C23—C24—H24 | 119.6 |
| C13—C1—H1 | 120.0 | C19—C24—H24 | 119.6 |
| C3—C2—C1 | 120.07 (14) | C30—C25—C26 | 118.69 (14) |
| C3—C2—H2 | 120.0 | C30—C25—C10 | 121.29 (14) |
| C1—C2—H2 | 120.0 | C26—C25—C10 | 119.99 (14) |
| C2—C3—C4 | 120.44 (15) | C27—C26—C25 | 120.88 (15) |
| C2—C3—H3 | 119.8 | C27—C26—H26 | 119.6 |
| C4—C3—H3 | 119.8 | C25—C26—H26 | 119.6 |
| C3—C4—C14 | 119.67 (14) | C26—C27—C28 | 120.10 (16) |
| C3—C4—H4 | 120.2 | C26—C27—H27 | 119.9 |
| C14—C4—H4 | 120.2 | C28—C27—H27 | 119.9 |
| C6—C5—C18 | 121.30 (15) | C29—C28—C27 | 119.40 (15) |
| C6—C5—H5 | 119.4 | C29—C28—H28 | 120.3 |
| C18—C5—H5 | 119.4 | C27—C28—H28 | 120.3 |
| C5—C6—C7 | 120.24 (14) | C28—C29—C30 | 120.62 (16) |

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| C5—C6—H6 | 119.9 | C28—C29—H29 | 119.7 |
| C7—C6—H6 | 119.9 | C30—C29—H29 | 119.7 |
| C8—C7—C6 | 120.24 (14) | C25—C30—C29 | 120.31 (15) |
| C8—C7—H7 | 119.9 | C25—C30—H30 | 119.8 |
| C6—C7—H7 | 119.9 | C29—C30—H30 | 119.8 |
| C7—C8—C17 | 121.05 (15) | C32—C31—C36 | 118.76 (14) |
| C7—C8—H8 | 119.5 | C32—C31—C11 | 121.69 (14) |
| C17—C8—H8 | 119.5 | C36—C31—C11 | 119.38 (14) |
| O1—C9—C19 | 102.17 (12) | C33—C32—C31 | 120.29 (15) |
| O1—C9—C13 | 105.08 (11) | C33—C32—H32 | 119.9 |
| C19—C9—C13 | 113.57 (12) | C31—C32—H32 | 119.9 |
| O1—C9—C15 | 105.00 (11) | C34—C33—C32 | 120.33 (16) |
| C19—C9—C15 | 120.06 (12) | C34—C33—H33 | 119.8 |
| C13—C9—C15 | 109.23 (12) | C32—C33—H33 | 119.8 |
| O2—C10—C14 | 105.51 (11) | C33—C34—C35 | 120.00 (15) |
| O2—C10—C25 | 102.69 (11) | C33—C34—H34 | 120.0 |
| C14—C10—C25 | 113.50 (12) | C35—C34—H34 | 120.0 |
| O2—C10—C16 | 104.75 (11) | C36—C35—C34 | 119.69 (15) |
| C14—C10—C16 | 109.49 (12) | C36—C35—H35 | 120.2 |
| C25—C10—C16 | 119.31 (12) | C34—C35—H35 | 120.2 |
| C15—C11—C17 | 118.87 (13) | C35—C36—C31 | 120.87 (15) |
| C15—C11—C31 | 125.89 (13) | C35—C36—H36 | 119.6 |
| C17—C11—C31 | 115.23 (13) | C31—C36—H36 | 119.6 |
| C16—C12—C18 | 118.85 (13) | C42—C37—C38 | 118.63 (14) |
| C16—C12—C37 | 126.41 (13) | C42—C37—C12 | 122.00 (14) |
| C18—C12—C37 | 114.73 (12) | C38—C37—C12 | 119.08 (14) |
| C1—C13—C14 | 119.72 (14) | C39—C38—C37 | 120.91 (15) |
| C1—C13—C9 | 127.11 (13) | C39—C38—H38 | 119.5 |
| C14—C13—C9 | 112.83 (13) | C37—C38—H38 | 119.5 |
| C13—C14—C4 | 119.85 (14) | C38—C39—C40 | 119.89 (16) |
| C13—C14—C10 | 113.04 (13) | C38—C39—H39 | 120.1 |
| C4—C14—C10 | 126.89 (14) | C40—C39—H39 | 120.1 |
| C11—C15—C16 | 120.99 (13) | C41—C40—C39 | 119.89 (15) |
| C11—C15—C9 | 127.08 (13) | C41—C40—H40 | 120.1 |
| C16—C15—C9 | 111.91 (12) | C39—C40—H40 | 120.1 |
| C12—C16—C15 | 120.80 (13) | C40—C41—C42 | 120.37 (15) |
| C12—C16—C10 | 127.73 (13) | C40—C41—H41 | 119.8 |
| C15—C16—C10 | 111.43 (12) | C42—C41—H41 | 119.8 |
| C18—C17—C8 | 118.69 (13) | C41—C42—C37 | 120.28 (16) |
| C18—C17—C11 | 120.12 (13) | C41—C42—H42 | 119.9 |
| C8—C17—C11 | 121.18 (14) | C37—C42—H42 | 119.9 |
| C17—C18—C5 | 118.48 (13) | O3—C43—C45 | 121.95 (18) |
| C17—C18—C12 | 120.33 (13) | O3—C43—C44 | 120.89 (18) |
| C5—C18—C12 | 121.18 (14) | C45—C43—C44 | 117.16 (17) |
| C24—C19—C20 | 118.35 (14) | C43—C44—H44A | 109.5 |
| C24—C19—C9 | 120.63 (14) | C43—C44—H44B | 109.5 |
| C20—C19—C9 | 120.95 (14) | H44A—C44—H44B | 109.5 |
| C21—C20—C19 | 120.63 (15) | C43—C44—H44C | 109.5 |
| C21—C20—H20 | 119.7 | H44A—C44—H44C | 109.5 |

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| C19—C20—H20 | 119.7 | H44B—C44—H44C | 109.5 |
| C22—C21—C20 | 120.20 (17) | C43—C45—H45A | 109.5 |
| C22—C21—H21 | 119.9 | C43—C45—H45B | 109.5 |
| C20—C21—H21 | 119.9 | H45A—C45—H45B | 109.5 |
| C21—C22—C23 | 119.73 (16) | C43—C45—H45C | 109.5 |
| C21—C22—H22 | 120.1 | H45A—C45—H45C | 109.5 |
| C23—C22—H22 | 120.1 | H45B—C45—H45C | 109.5 |
| C24—C23—C22 | 120.14 (16) | | |

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| C9—O1—O2—C10 | 2.79 (14) | C8—C17—C18—C5 | 1.1 (2) |
| C13—C1—C2—C3 | −3.2 (2) | C11—C17—C18—C5 | −179.82 (14) |
| C1—C2—C3—C4 | 0.4 (2) | C8—C17—C18—C12 | −177.41 (14) |
| C2—C3—C4—C14 | 3.1 (2) | C11—C17—C18—C12 | 1.7 (2) |
| C18—C5—C6—C7 | −0.2 (3) | C6—C5—C18—C17 | −0.6 (2) |
| C5—C6—C7—C8 | 0.4 (3) | C6—C5—C18—C12 | 177.93 (15) |
| C6—C7—C8—C17 | 0.2 (3) | C16—C12—C18—C17 | −0.1 (2) |
| O2—O1—C9—C19 | −177.18 (10) | C37—C12—C18—C17 | −179.42 (14) |
| O2—O1—C9—C13 | −58.38 (13) | C16—C12—C18—C5 | −178.62 (14) |
| O2—O1—C9—C15 | 56.79 (13) | C37—C12—C18—C5 | 2.1 (2) |
| O1—O2—C10—C14 | 55.15 (13) | O1—C9—C19—C24 | 168.04 (13) |
| O1—O2—C10—C25 | 174.28 (10) | C13—C9—C19—C24 | 55.43 (18) |
| O1—O2—C10—C16 | −60.39 (13) | C15—C9—C19—C24 | −76.45 (18) |
| C2—C1—C13—C14 | 2.4 (2) | O1—C9—C19—C20 | −8.93 (18) |
| C2—C1—C13—C9 | 175.14 (14) | C13—C9—C19—C20 | −121.53 (15) |
| O1—C9—C13—C1 | −117.38 (15) | C15—C9—C19—C20 | 106.58 (16) |
| C19—C9—C13—C1 | −6.5 (2) | C24—C19—C20—C21 | −1.8 (2) |
| C15—C9—C13—C1 | 130.43 (15) | C9—C19—C20—C21 | 175.27 (13) |
| O1—C9—C13—C14 | 55.81 (15) | C19—C20—C21—C22 | 0.4 (2) |
| C19—C9—C13—C14 | 166.65 (12) | C20—C21—C22—C23 | 1.5 (2) |
| C15—C9—C13—C14 | −56.38 (16) | C21—C22—C23—C24 | −1.9 (2) |
| C1—C13—C14—C4 | 1.1 (2) | C22—C23—C24—C19 | 0.5 (2) |
| C9—C13—C14—C4 | −172.63 (13) | C20—C19—C24—C23 | 1.4 (2) |
| C1—C13—C14—C10 | 176.12 (13) | C9—C19—C24—C23 | −175.69 (14) |
| C9—C13—C14—C10 | 2.37 (17) | O2—C10—C25—C30 | 9.34 (17) |
| C3—C4—C14—C13 | −3.8 (2) | C14—C10—C25—C30 | 122.72 (15) |
| C3—C4—C14—C10 | −178.06 (14) | C16—C10—C25—C30 | −105.86 (16) |
| O2—C10—C14—C13 | −58.71 (15) | O2—C10—C25—C26 | −168.74 (12) |
| C25—C10—C14—C13 | −170.39 (12) | C14—C10—C25—C26 | −55.35 (18) |
| C16—C10—C14—C13 | 53.53 (16) | C16—C10—C25—C26 | 76.06 (18) |
| O2—C10—C14—C4 | 115.86 (16) | C30—C25—C26—C27 | −0.4 (2) |
| C25—C10—C14—C4 | 4.2 (2) | C10—C25—C26—C27 | 177.76 (13) |
| C16—C10—C14—C4 | −131.89 (15) | C25—C26—C27—C28 | 0.6 (2) |
| C17—C11—C15—C16 | 0.0 (2) | C26—C27—C28—C29 | −0.1 (2) |
| C31—C11—C15—C16 | −178.71 (14) | C27—C28—C29—C30 | −0.5 (2) |
| C17—C11—C15—C9 | −177.93 (14) | C26—C25—C30—C29 | −0.2 (2) |
| C31—C11—C15—C9 | 3.4 (3) | C10—C25—C30—C29 | −178.33 (13) |
| O1—C9—C15—C11 | 118.96 (16) | C28—C29—C30—C25 | 0.6 (2) |
| C19—C9—C15—C11 | 4.9 (2) | C15—C11—C31—C32 | −79.6 (2) |
| C13—C9—C15—C11 | −128.79 (16) | C17—C11—C31—C32 | 101.72 (17) |

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| O1—C9—C15—C16 | −59.09 (15) | C15—C11—C31—C36 | 105.37 (18) |
| C19—C9—C15—C16 | −173.12 (13) | C17—C11—C31—C36 | −73.35 (18) |
| C13—C9—C15—C16 | 53.16 (16) | C36—C31—C32—C33 | −2.7 (2) |
| C18—C12—C16—C15 | −1.5 (2) | C11—C31—C32—C33 | −177.81 (13) |
| C37—C12—C16—C15 | 177.73 (14) | C31—C32—C33—C34 | 1.4 (2) |
| C18—C12—C16—C10 | 176.09 (14) | C32—C33—C34—C35 | 0.6 (2) |
| C37—C12—C16—C10 | −4.7 (3) | C33—C34—C35—C36 | −1.3 (2) |
| C11—C15—C16—C12 | 1.6 (2) | C34—C35—C36—C31 | 0.0 (2) |
| C9—C15—C16—C12 | 179.76 (13) | C32—C31—C36—C35 | 2.0 (2) |
| C11—C15—C16—C10 | −176.35 (13) | C11—C31—C36—C35 | 177.23 (13) |
| C9—C15—C16—C10 | 1.84 (17) | C16—C12—C37—C42 | 79.8 (2) |
| O2—C10—C16—C12 | −120.29 (16) | C18—C12—C37—C42 | −100.95 (16) |
| C14—C10—C16—C12 | 126.97 (16) | C16—C12—C37—C38 | −106.47 (18) |
| C25—C10—C16—C12 | −6.2 (2) | C18—C12—C37—C38 | 72.76 (18) |
| O2—C10—C16—C15 | 57.46 (15) | C42—C37—C38—C39 | −1.5 (2) |
| C14—C10—C16—C15 | −55.28 (16) | C12—C37—C38—C39 | −175.42 (14) |
| C25—C10—C16—C15 | 171.56 (13) | C37—C38—C39—C40 | 0.5 (2) |
| C7—C8—C17—C18 | −1.0 (2) | C38—C39—C40—C41 | 0.1 (2) |
| C7—C8—C17—C11 | 180.00 (15) | C39—C40—C41—C42 | 0.5 (2) |
| C15—C11—C17—C18 | −1.5 (2) | C40—C41—C42—C37 | −1.6 (2) |
| C31—C11—C17—C18 | 177.27 (14) | C38—C37—C42—C41 | 2.0 (2) |
| C15—C11—C17—C8 | 177.49 (15) | C12—C37—C42—C41 | 175.78 (13) |
| C31—C11—C17—C8 | −3.7 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C28—H28···O3$^{i}$ | 0.95 | 2.53 | 3.437 (2) | 159 |
| C35—H35···O1$^{ii}$ | 0.95 | 2.59 | 3.408 (2) | 144 |

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