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3,8-Dimethylacenaphthylene-1,2-dione

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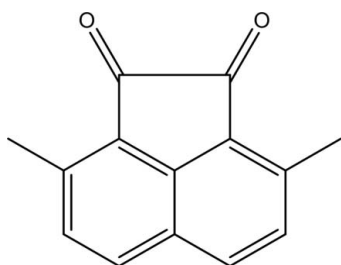
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.052; wR factor = 0.148; data-to-parameter ratio = 12.5.

In the title compound, $\text{C}_{14}\text{H}_{10}\text{O}_2$, the acenaphthenequinone core is essentially planar, with an r.m.s. deviation of 0.0140 Å. In the crystal, molecules are connected by π - π stacking interactions [centroid-centroid distances = 3.766 (3), 3.839 (3) and 3.857 (3) Å], forming columns parallel to the a axis.

Related literature

For the synthesis and applications of corannulene (systematic name: dibenzo[*ghi,mno*]fluoranthene) and its derivatives, see: Wu & Siegel (2006); Tsefrikas & Scott (2006); Sygula (2011); Zabula *et al.* (2011); Barth & Lawton (1966); Scott *et al.* (1997). For the synthesis of the title compound, see: Guillermet *et al.* (2009); Seiders *et al.* (1999); Mori *et al.* (2007). For the structure of related compounds, see: Abdourazak *et al.* (1994); Mochida & Yoza (2010).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_{10}\text{O}_2$ $M_r = 210.22$

Triclinic, $P\bar{1}$
 $a = 7.5415$ (8) Å
 $b = 8.5562$ (11) Å
 $c = 9.8925$ (12) Å
 $\alpha = 67.891$ (11)°
 $\beta = 88.310$ (9)°
 $\gamma = 63.998$ (11)°

$V = 524.45$ (13) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.42 \times 0.35 \times 0.35$ mm

Data collection

Agilent Xcalibur Eos diffractometer
Absorption correction: multi-scan
(*CrysAlis PRO*; Agilent, 2010)
 $T_{\min} = 0.964$, $T_{\max} = 1.000$

3865 measured reflections
1844 independent reflections
1247 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.148$
 $S = 1.09$
1844 reflections

147 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.17$ e Å⁻³
 $\Delta\rho_{\min} = -0.17$ e Å⁻³

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ22626).

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supporting information

Acta Cryst. (2011). E67, o2130 [doi:10.1107/S1600536811028479]

3,8-Dimethylacenaphthylene-1,2-dione

Shu-hua He

S1. Comment

Corannulene (IUPAC name: dibenzo[*ghi,mno*]fluoranthene) and its derivatives have triggered intense interest due to their unusual bowl-formed structure (Wu & Siegel, 2006; Tsefrikas & Scott, 2006). Recently, a number of novel corannulene derivatives with excellent optical properties have been reported (Sygula, 2011; Zabula *et al.*, 2011). Up to now, different synthetic methods have been reported (Barth & Lawton, 1966; Scott *et al.*, 1997). The title compound, the synthesis of which has been reported in literatures (Guillermet *et al.*, 2009; Seiders *et al.*, 1999; Mori *et al.*, 2007), is an important intermediate in synthesizing corannulene and its derivatives. Based on the importance of the relationship between structures and properties, the crystal structure of the title compound is reported herein.

The title molecule (Fig. 1) possesses a *pseudo*-mirror plane and is substantially planar, with a r.m.s. deviation of 0.0140 Å and maximum displacement of 0.026 (3) Å for atom C8. The dihedral angles between the C1–C5 five membered ring and the C3–C4/C9–C12 and C4–C9 benzene rings are 1.42 (9) and 0.81 (9)°, respectively. The C13–C6–C5–C1, C13–C6–C7–C8, C14–C12–C3–C2 and C14–C12–C11–C10 torsion angles are 3.6 (5), 178.4 (3), 0.4 (5) and 178.5 (4)°, respectively. As already observed in related compounds (Abdourazak *et al.*, 1994; Mochida & Yoza, 2010), the C1–C2 bond length (1.569 (4) Å) is remarkably longer than expected for a C(*sp*²)–C(*sp*²) bond, and the bond angles involving the carbonyl groups (O1–C1–C5 = 130.6 (3)°, O1–C1–C2 = 123.5 (2)°, C6–C5–C1 = 133.3 (2)°) are larger than the ideal value of 120°. In the crystal (Fig. 2), molecules are linked into columns parallel to the *a* axis by π – π stacking interactions [Cg1...Cg2ⁱ = 3.766 (3) Å; Cg2...Cg2ⁱ = 3.839 (3) Å; Cg2...Cg2ⁱⁱ = 3.857 (3) Å; Cg1 and Cg2 are the centroids of the C3–C4/C9–C12 and C4–C9 rings, respectively. Symmetry codes: (i) 2-x, 1-y, 2-z; (ii) 1-x, 1-y, 2-z].

S2. Experimental

To a mixture of aluminium tribromide (10 g, 37.5 mmol) in CH₂Cl₂ (200 ml) was added dropwise a solution of 2,7-dimethylnaphthalene (2.6 g, 16.6 mmol) and oxalylchloride (2.2 ml, 26.0 mmol) in CH₂Cl₂ (150 ml) at about 253 K over 15 min. The mixture was stirred vigorously at 253 K for additional 6 h and then quenched carefully by pouring into 500 ml ice water. After 30 min, the organic layer was washed with water, dried over MgSO₄, filtered and evaporated. The residue was dissolved in a minimal amount of CH₂Cl₂ and eluted with CH₂Cl₂/hexane (1:1 v/v) through an alumina column to give the title compound as a bright yellow solid (1.26 g, 37% yield, m.p. 478–480 K). Bright yellow single crystals suitable for X-ray diffraction were obtained at room temperature by slow evaporation of a CH₂Cl₂ solution over a period of several days.

S3. Refinement

All H atoms were positioned geometrically (C–H = 0.93–0.96 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

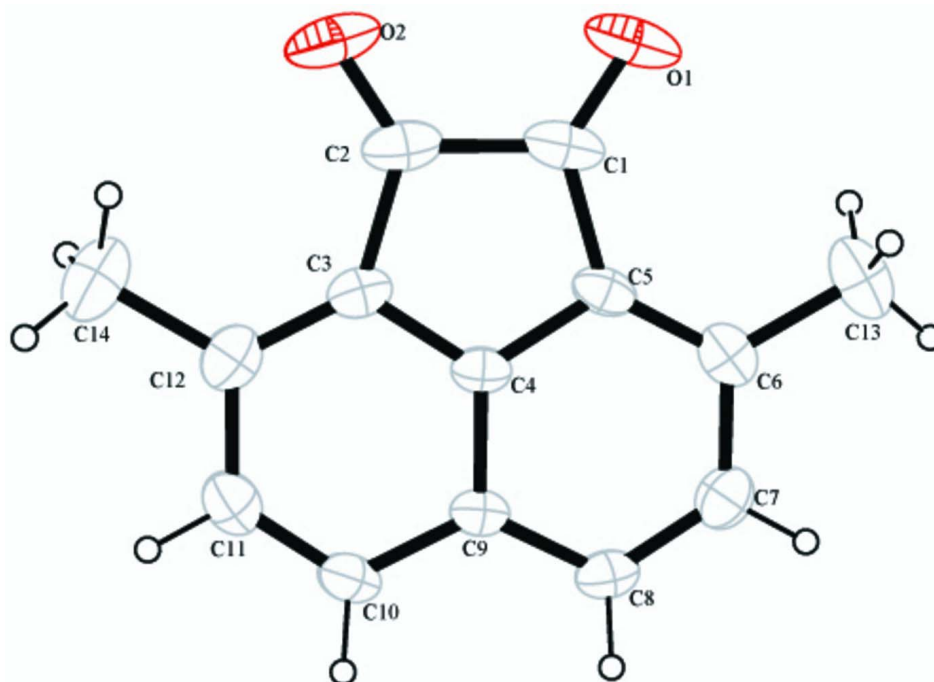


Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

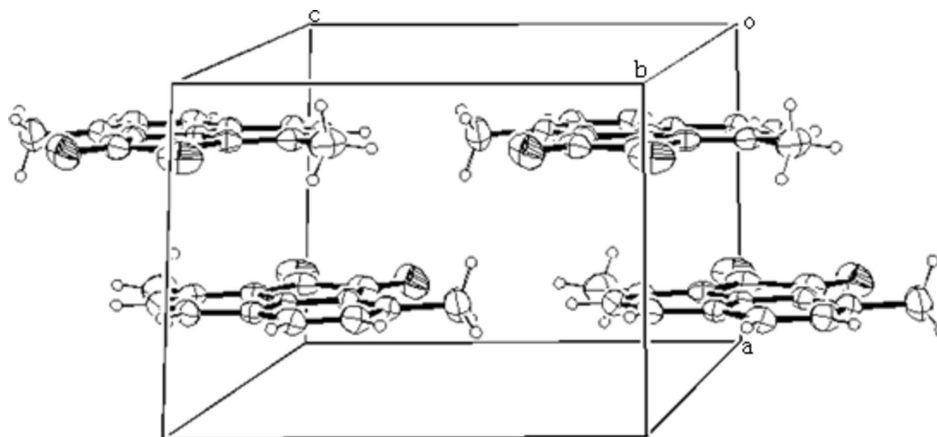


Figure 2

Packing diagram of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

3,8-Dimethylnaphthalene-1,2-dione

Crystal data

$C_{14}H_{10}O_2$

$M_r = 210.22$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.5415$ (8) Å

$b = 8.5562$ (11) Å

$c = 9.8925$ (12) Å

$\alpha = 67.891$ (11)°

$\beta = 88.310$ (9)°

$\gamma = 63.998$ (11)°

$V = 524.45$ (13) Å³

$Z = 2$

$F(000) = 220$

$D_x = 1.331$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å

Cell parameters from 1026 reflections

$\theta = 2.9\text{--}29.4^\circ$
 $\mu = 0.09\text{ mm}^{-1}$
 $T = 293\text{ K}$

Block, yellow
 $0.42 \times 0.35 \times 0.35\text{ mm}$

Data collection

Agilent Xcalibur Eos
 diffractometer
 Radiation source: Enhance (Mo) X-ray Source
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan
 (CrysAlis PRO; Agilent, 2010)
 $T_{\min} = 0.964$, $T_{\max} = 1.000$

3865 measured reflections
 1844 independent reflections
 1247 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.9^\circ$
 $h = -8 \rightarrow 8$
 $k = -9 \rightarrow 10$
 $l = -11 \rightarrow 11$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.148$
 $S = 1.09$
 1844 reflections
 147 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
 $w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 0.102P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.17\text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.17\text{ e \AA}^{-3}$

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 | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|------------|------------|------------|----------------------------------|
| O1 | 0.7750 (3) | 0.0525 (3) | 1.0053 (3) | 0.0920 (7) |
| O2 | 0.7510 (3) | 0.2516 (3) | 0.6896 (3) | 0.0968 (8) |
| C1 | 0.7662 (3) | 0.2086 (3) | 0.9497 (3) | 0.0640 (8) |
| C2 | 0.7529 (3) | 0.3165 (4) | 0.7793 (3) | 0.0652 (7) |
| C3 | 0.7426 (3) | 0.4999 (3) | 0.7582 (3) | 0.0507 (6) |
| C4 | 0.7488 (3) | 0.5024 (3) | 0.8994 (2) | 0.0425 (5) |
| C5 | 0.7647 (3) | 0.3356 (3) | 1.0170 (3) | 0.0480 (6) |
| C6 | 0.7721 (3) | 0.3212 (3) | 1.1597 (3) | 0.0545 (7) |
| C7 | 0.7594 (3) | 0.4792 (4) | 1.1814 (3) | 0.0584 (7) |
| H7 | 0.7629 | 0.4727 | 1.2774 | 0.070* |
| C8 | 0.7422 (3) | 0.6419 (3) | 1.0686 (3) | 0.0531 (6) |
| H8 | 0.7337 | 0.7421 | 1.0895 | 0.064* |
| C9 | 0.7374 (3) | 0.6586 (3) | 0.9216 (3) | 0.0446 (6) |

| | | | | |
|------|------------|------------|------------|-------------|
| C10 | 0.7241 (3) | 0.8142 (3) | 0.7938 (3) | 0.0547 (6) |
| H10 | 0.7177 | 0.9209 | 0.8026 | 0.066* |
| C11 | 0.7205 (4) | 0.8089 (4) | 0.6583 (3) | 0.0616 (7) |
| H11 | 0.7125 | 0.9134 | 0.5769 | 0.074* |
| C12 | 0.7283 (3) | 0.6538 (4) | 0.6344 (3) | 0.0592 (7) |
| C13 | 0.7965 (4) | 0.1452 (4) | 1.2891 (3) | 0.0820 (9) |
| H13A | 0.7773 | 0.1705 | 1.3765 | 0.123* |
| H13B | 0.9284 | 0.0439 | 1.3032 | 0.123* |
| H13C | 0.6992 | 0.1088 | 1.2703 | 0.123* |
| C14 | 0.7185 (5) | 0.6599 (5) | 0.4806 (3) | 0.0916 (10) |
| H14A | 0.7763 | 0.7373 | 0.4208 | 0.137* |
| H14B | 0.5815 | 0.7129 | 0.4380 | 0.137* |
| H14C | 0.7915 | 0.5333 | 0.4849 | 0.137* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| O1 | 0.0767 (13) | 0.0459 (11) | 0.160 (2) | -0.0320 (10) | 0.0113 (13) | -0.0430 (12) |
| O2 | 0.0918 (15) | 0.0941 (15) | 0.1333 (19) | -0.0328 (12) | 0.0040 (13) | -0.0859 (16) |
| C1 | 0.0407 (13) | 0.0435 (14) | 0.112 (2) | -0.0185 (11) | 0.0053 (14) | -0.0368 (15) |
| C2 | 0.0447 (14) | 0.0609 (16) | 0.102 (2) | -0.0178 (12) | 0.0048 (13) | -0.0525 (17) |
| C3 | 0.0407 (12) | 0.0524 (14) | 0.0655 (16) | -0.0182 (11) | 0.0054 (11) | -0.0340 (13) |
| C4 | 0.0320 (11) | 0.0403 (12) | 0.0601 (15) | -0.0167 (9) | 0.0068 (10) | -0.0251 (11) |
| C5 | 0.0362 (12) | 0.0374 (12) | 0.0707 (17) | -0.0182 (10) | 0.0073 (11) | -0.0204 (12) |
| C6 | 0.0374 (13) | 0.0507 (14) | 0.0635 (17) | -0.0185 (11) | 0.0077 (11) | -0.0134 (12) |
| C7 | 0.0515 (14) | 0.0681 (17) | 0.0553 (16) | -0.0239 (13) | 0.0090 (12) | -0.0292 (14) |
| C8 | 0.0517 (14) | 0.0504 (14) | 0.0665 (17) | -0.0219 (12) | 0.0078 (12) | -0.0345 (13) |
| C9 | 0.0379 (11) | 0.0395 (12) | 0.0596 (15) | -0.0178 (9) | 0.0055 (10) | -0.0233 (11) |
| C10 | 0.0543 (14) | 0.0414 (13) | 0.0690 (18) | -0.0252 (11) | 0.0020 (12) | -0.0186 (12) |
| C11 | 0.0575 (15) | 0.0549 (15) | 0.0617 (18) | -0.0273 (13) | 0.0039 (12) | -0.0108 (13) |
| C12 | 0.0447 (14) | 0.0706 (17) | 0.0581 (16) | -0.0205 (12) | 0.0043 (11) | -0.0289 (14) |
| C13 | 0.0662 (18) | 0.0680 (18) | 0.080 (2) | -0.0287 (15) | 0.0118 (15) | -0.0004 (16) |
| C14 | 0.087 (2) | 0.114 (3) | 0.064 (2) | -0.031 (2) | 0.0055 (16) | -0.0442 (19) |

Geometric parameters (Å, °)

| | | | |
|--------|-----------|----------|-----------|
| O1—C1 | 1.209 (3) | C8—H8 | 0.9300 |
| O2—C2 | 1.214 (3) | C8—C9 | 1.407 (3) |
| C1—C2 | 1.569 (4) | C9—C10 | 1.416 (3) |
| C1—C5 | 1.468 (3) | C10—H10 | 0.9300 |
| C2—C3 | 1.470 (3) | C10—C11 | 1.360 (3) |
| C3—C4 | 1.407 (3) | C11—H11 | 0.9300 |
| C3—C12 | 1.384 (3) | C11—C12 | 1.410 (3) |
| C4—C5 | 1.419 (3) | C12—C14 | 1.506 (4) |
| C4—C9 | 1.401 (3) | C13—H13A | 0.9600 |
| C5—C6 | 1.370 (3) | C13—H13B | 0.9600 |
| C6—C7 | 1.410 (3) | C13—H13C | 0.9600 |
| C6—C13 | 1.504 (3) | C14—H14A | 0.9600 |

| | | | |
|---------------|--------------|-----------------|--------------|
| C7—H7 | 0.9300 | C14—H14B | 0.9600 |
| C7—C8 | 1.372 (3) | C14—H14C | 0.9600 |
| O1—C1—C2 | 123.5 (2) | C4—C9—C8 | 116.2 (2) |
| O1—C1—C5 | 130.6 (3) | C4—C9—C10 | 116.3 (2) |
| C5—C1—C2 | 105.9 (2) | C8—C9—C10 | 127.5 (2) |
| O2—C2—C1 | 123.5 (2) | C9—C10—H10 | 119.7 |
| O2—C2—C3 | 130.3 (3) | C11—C10—C9 | 120.5 (2) |
| C3—C2—C1 | 106.2 (2) | C11—C10—H10 | 119.7 |
| C4—C3—C2 | 106.6 (2) | C10—C11—H11 | 118.2 |
| C12—C3—C2 | 133.0 (2) | C10—C11—C12 | 123.6 (2) |
| C12—C3—C4 | 120.4 (2) | C12—C11—H11 | 118.2 |
| C3—C4—C5 | 114.79 (19) | C3—C12—C11 | 116.6 (2) |
| C9—C4—C3 | 122.5 (2) | C3—C12—C14 | 122.7 (2) |
| C9—C4—C5 | 122.7 (2) | C11—C12—C14 | 120.7 (3) |
| C4—C5—C1 | 106.5 (2) | C6—C13—H13A | 109.5 |
| C6—C5—C1 | 133.3 (2) | C6—C13—H13B | 109.5 |
| C6—C5—C4 | 120.2 (2) | C6—C13—H13C | 109.5 |
| C5—C6—C7 | 116.9 (2) | H13A—C13—H13B | 109.5 |
| C5—C6—C13 | 122.5 (2) | H13A—C13—H13C | 109.5 |
| C7—C6—C13 | 120.6 (2) | H13B—C13—H13C | 109.5 |
| C6—C7—H7 | 118.3 | C12—C14—H14A | 109.5 |
| C8—C7—C6 | 123.5 (2) | C12—C14—H14B | 109.5 |
| C8—C7—H7 | 118.3 | C12—C14—H14C | 109.5 |
| C7—C8—H8 | 119.7 | H14A—C14—H14B | 109.5 |
| C7—C8—C9 | 120.5 (2) | H14A—C14—H14C | 109.5 |
| C9—C8—H8 | 119.7 | H14B—C14—H14C | 109.5 |
| O1—C1—C2—O2 | -0.2 (4) | C4—C3—C12—C14 | -179.2 (2) |
| O1—C1—C2—C3 | 179.3 (2) | C4—C5—C6—C7 | 1.2 (3) |
| O1—C1—C5—C4 | -179.0 (2) | C4—C5—C6—C13 | -177.7 (2) |
| O1—C1—C5—C6 | -0.1 (4) | C4—C9—C10—C11 | 0.7 (3) |
| O2—C2—C3—C4 | 179.5 (3) | C5—C1—C2—O2 | 179.9 (2) |
| O2—C2—C3—C12 | -0.2 (4) | C5—C1—C2—C3 | -0.5 (2) |
| C1—C2—C3—C4 | 0.0 (2) | C5—C4—C9—C8 | -0.1 (3) |
| C1—C2—C3—C12 | -179.7 (2) | C5—C4—C9—C10 | 179.39 (18) |
| C1—C5—C6—C7 | -177.6 (2) | C5—C6—C7—C8 | -0.6 (3) |
| C1—C5—C6—C13 | 3.5 (4) | C6—C7—C8—C9 | -0.4 (3) |
| C2—C1—C5—C4 | 0.8 (2) | C7—C8—C9—C4 | 0.7 (3) |
| C2—C1—C5—C6 | 179.7 (2) | C7—C8—C9—C10 | -178.7 (2) |
| C2—C3—C4—C5 | 0.6 (2) | C8—C9—C10—C11 | -179.8 (2) |
| C2—C3—C4—C9 | -178.55 (18) | C9—C4—C5—C1 | 178.21 (18) |
| C2—C3—C12—C11 | 179.6 (2) | C9—C4—C5—C6 | -0.9 (3) |
| C2—C3—C12—C14 | 0.4 (4) | C9—C10—C11—C12 | 0.4 (4) |
| C3—C4—C5—C1 | -1.0 (2) | C10—C11—C12—C3 | -0.7 (4) |
| C3—C4—C5—C6 | 179.97 (18) | C10—C11—C12—C14 | 178.5 (2) |
| C3—C4—C9—C8 | 178.99 (19) | C12—C3—C4—C5 | -179.64 (19) |
| C3—C4—C9—C10 | -1.5 (3) | C12—C3—C4—C9 | 1.2 (3) |

C4—C3—C12—C11

0.0 (3)

C13—C6—C7—C8

178.3 (2)
