

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

 2-Bromo-*p*-terphenyl

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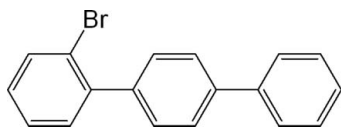
Received 12 February 2010; accepted 26 February 2010

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.034; wR factor = 0.092; data-to-parameter ratio = 20.4.

In the title compound, $\text{C}_{18}\text{H}_{13}\text{Br}$, the dihedral angles between the mean planes of the central benzene ring and the mean planes of the outer phenyl and bromophenyl rings are $33.47(8)$ and $66.35(8)^\circ$, respectively. In the crystal, weak $\text{C}-\text{H}\cdots\pi$ and intermolecular $\text{Br}\cdots\text{Br}$ [$3.5503(15)$ Å] interactions contribute to the stabilization of the packing.

Related literature

For the synthesis, see: France *et al.* (1938); Tadashi *et al.* (1962). For the Suzuki coupling reaction, see: Miyaura & Suzuki (1995). For cross-coupling reactions of *o*-halogenated arenes, see: Ishikawa & Manabe (2007). For organic light-emitting diodes, see: Kim *et al.* (2008). For related structures, see: Jones *et al.* (2005); Liang (2008); MacNeil & Decken (1999); Politzer *et al.* (2007).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{13}\text{Br}$ $V = 2810(2)$ Å³
 $M_r = 309.19$ $Z = 8$
 Monoclinic, $C2/c$ $\text{Mo K}\alpha$ radiation
 $a = 27.039(10)$ Å $\mu = 2.91$ mm⁻¹
 $b = 7.597(3)$ Å $T = 293$ K
 $c = 18.907(7)$ Å $0.30 \times 0.25 \times 0.20$ mm
 $\beta = 133.650(5)^\circ$

Data collection

Bruker SMART CCD area-detector diffractometer 13933 measured reflections
 Absorption correction: multi-scan (SADABS; Sheldrick, 1999) 3503 independent reflections
 $T_{\min} = 0.476$, $T_{\max} = 0.594$ 2246 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$ 172 parameters
 $wR(F^2) = 0.092$ H-atom parameters constrained
 $S = 1.02$ $\Delta\rho_{\max} = 0.43$ e Å⁻³
 3503 reflections $\Delta\rho_{\min} = -0.65$ e Å⁻³

Table 1

 C—H $\cdots\pi$ interactions (Å, °).

Cg2 and Cg3 are the centroids of the C7–C12 and C13–C18 rings, respectively.

D—H \cdots A	D—H	H \cdots A	D \cdots A	D—H \cdots A
C2—H2 \cdots Cg3 ⁱ	0.93	2.84	3.778 (4)	148
C14—H14 \cdots Cg2 ⁱⁱ	0.93	2.97	3.658 (5)	147

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT-Plus (Bruker, 2000); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

This research was supported by the Basic Science Research Program through the National Research Foundation of Korea (NRF) and funded by the Ministry of Education, Science and Technology. (2009-0072468)

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2023).

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supporting information

Acta Cryst. (2010). E66, o743 [doi:10.1107/S1600536810007348]

2-Bromo-*p*-terphenyl

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S1. Comment

Palladium-catalyzed cross-coupling reactions of aryl halides with arylboronic acids, often referred as Suzuki coupling reactions, are versatile synthetic methods for the preparation of unsymmetrical biaryls. The Suzuki coupling reactions have been applied extensively in the synthesis of natural products, nucleoside analogues, and pharmaceuticals (Miyaura & Suzuki, 1995). Cross-coupling reactions of *o*-halogenated arenes are very useful synthetically, if the halogen atom is converted to other functional groups, such as carbazole, anthracene and fluorene (Ishikawa & Manabe, 2007). Anthracene based terphenyl derivatives are widely used as emitting and/or host materials in organic light-emitting diodes (OLEDs) (Kim *et al.*, 2008). To be good host materials in OLEDs, the host must have larger energy gap between the HOMO (Highest Occupied Molecular Orbital) and the LUMO (Lowest Unoccupied Molecular orbital) than in a dopant, because energy transfer occurs from host to dopant.

The introduction of substituents at the *ortho*-position of biaryl and terphenyl groups is often used in the preparation of an efficient host with a wide band gap, because the substituents suppress effective π -conjugation throughout the whole molecule. Therefore, the structures of biaryl and terphenyl derivatives bearing a halogen atom at the *ortho*-position are attractive as good precursors to materials oriented chemists and physicists. The title compound, C₁₈H₁₃Br, was synthesized by the Pd-catalyzed cross coupling of 4-biphenylboronic acid with 1-bromo-2-iodobenzene in the presence of base (Na₂CO₃).

The dihedral angles between the mean planes of the central phenyl ring (C7-C12) and the mean planes of the outer phenyl (C13-C18) and the brominated phenyl (C1-C6) rings, are 33.47 (8)° and 66.35 (8)°, respectively (Fig. 1). All bond lengths and bond angles are normal and comparable to those observed in similar structures (MacNeil & Decken, 1999; Jones *et al.*, 2005).

Weak C–H \cdots Cg π -ring interactions are observed [C2–H2 \cdots Cg3; H2 \cdots Cg3 = \AA ; C2–H2 \cdots Cg = 148°, C2 \cdots Cg3–H2 = 3.778 (4) \AA ; 1/2+x, 1/2-y, 1/2+z and C14–H14 \cdots Cg2; H14 \cdots Cg2 = 2.97 \AA ; C14–H14 \cdots Cg2 = 147°, C14 \cdots Cg2–H14 = 3.658 (5) \AA ; 1-x, y, 1/2-z; where Cg2 and Cg3 are the centroids of C7—C12 and C13—C18, respectively] (Fig. 2). Weak Br1 \cdots Br1 interactions also exist (3.5503 (15) \AA ; Politzer *et al.*, 2007; Liang, 2008) and along with C–H \cdots Cg π -ring interactions contribute to the stabilization of crystal packing.

S2. Experimental

A mixture of 4-biphenylboronic acid (0.1 mol), 1-bromo-2-iodobenzene (0.1 mol), Na₂CO₃ (0.6 mol, 2M in H₂O), and Pd(PPh₃)₄ (5% mol) was refluxed for 12 h. After being cooled to room temperature, the reaction mixture was quenched by water. The aqueous layer was extracted with CH₂Cl₂, and the combined organic layers were sequentially washed with saturated aqueous NaCl (20 mL), dried with MgSO₄, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography using CH₂Cl₂ and hexane to give the titled compound as a colorless solid. Analytical data (France, *et al.*, 1938; Tadashi, *et al.*, 1962). ¹H NMR (CDCl₃, 300 MHz): 7.65 (m, 5H), 7.51 (m, 4H), 7.40

(m, 3H), 7.21 (m, 1H); MS(EI, m/z): 309 [M^+]. Slow evaporation of CH_2Cl_2 gave suitable single crystals for X-ray analysis.

S3. Refinement

All H-atoms were positioned geometrically and refined using a riding model with $\text{C-H} = 0.93 \text{ \AA}$, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

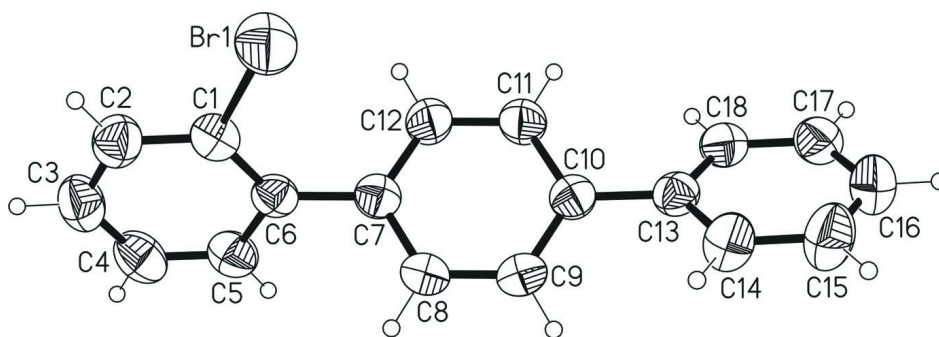


Figure 1

Molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

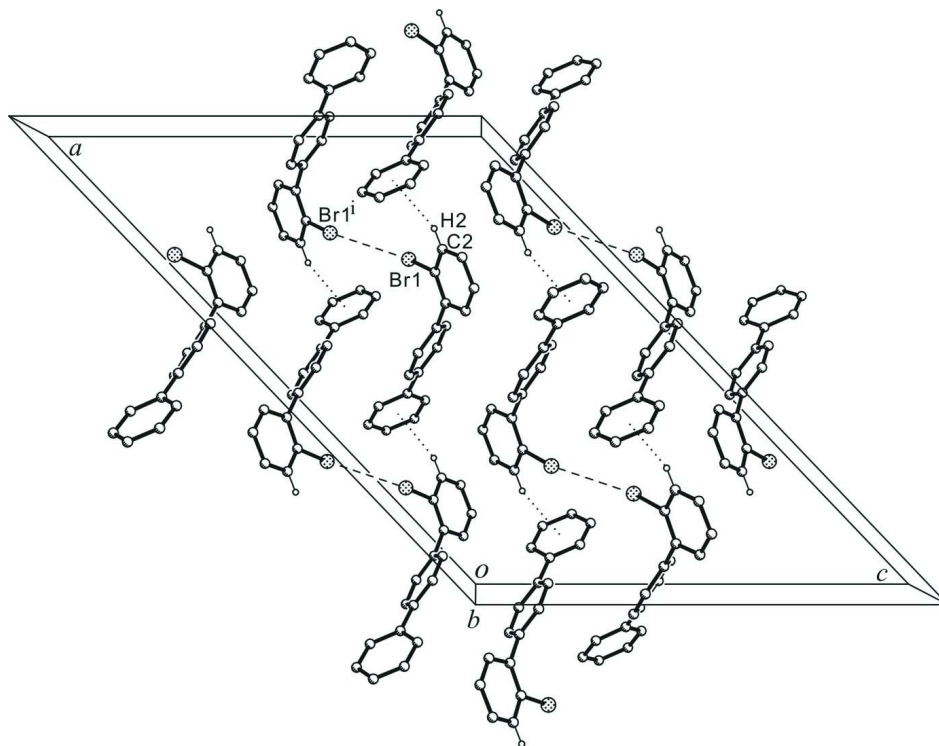


Figure 2

Weak $\text{Br}\cdots\text{Br}$ and $\text{C-H}\cdots\text{Cg}$ π -ring interactions (dashed lines) in the title compound.

2-Bromo-*p*-terphenyl*Crystal data*C₁₈H₁₃Br $M_r = 309.19$ Monoclinic, *C2/c*

Hall symbol: -C 2yc

 $a = 27.039 (10) \text{ \AA}$ $b = 7.597 (3) \text{ \AA}$ $c = 18.907 (7) \text{ \AA}$ $\beta = 133.650 (5)^\circ$ $V = 2810 (2) \text{ \AA}^3$ $Z = 8$ $F(000) = 1248$ $D_x = 1.462 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3654 reflections

 $\theta = 2.1\text{--}28.4^\circ$ $\mu = 2.91 \text{ mm}^{-1}$ $T = 293 \text{ K}$

Block, colorless

 $0.30 \times 0.25 \times 0.20 \text{ mm}$ *Data collection*Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scansAbsorption correction: multi-scan
(*SADABS*; Sheldrick, 1999) $T_{\min} = 0.476$, $T_{\max} = 0.594$

13933 measured reflections

3503 independent reflections

2246 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.028$ $\theta_{\text{max}} = 28.4^\circ$, $\theta_{\text{min}} = 2.1^\circ$ $h = -34 \rightarrow 36$ $k = -9 \rightarrow 10$ $l = -25 \rightarrow 25$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.034$ $wR(F^2) = 0.092$ $S = 1.02$

3503 reflections

172 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 2.6031P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.001$ $\Delta\rho_{\text{max}} = 0.43 \text{ e \AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.65 \text{ e \AA}^{-3}$ *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.

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.721104 (15)	0.25216 (4)	0.56047 (2)	0.07170 (13)
C1	0.69010 (12)	0.4341 (3)	0.59063 (16)	0.0458 (5)
C2	0.73645 (13)	0.5631 (3)	0.65377 (18)	0.0565 (6)
H2	0.7807	0.5590	0.6797	0.068*

C3	0.71685 (15)	0.6982 (3)	0.67834 (19)	0.0613 (7)
H3	0.7478	0.7861	0.7210	0.074*
C4	0.65143 (16)	0.7030 (3)	0.6398 (2)	0.0616 (7)
H4	0.6384	0.7930	0.6575	0.074*
C5	0.60498 (13)	0.5756 (3)	0.57514 (18)	0.0552 (6)
H5	0.5606	0.5819	0.5489	0.066*
C6	0.62281 (11)	0.4369 (3)	0.54788 (15)	0.0434 (5)
C7	0.57071 (12)	0.3039 (3)	0.47552 (17)	0.0446 (5)
C8	0.51546 (12)	0.3516 (3)	0.37957 (17)	0.0519 (6)
H8	0.5104	0.4684	0.3609	0.062*
C9	0.46779 (12)	0.2289 (3)	0.31111 (18)	0.0522 (6)
H9	0.4310	0.2648	0.2472	0.063*
C10	0.47356 (11)	0.0529 (3)	0.33573 (16)	0.0441 (5)
C11	0.52859 (12)	0.0067 (3)	0.43275 (16)	0.0520 (6)
H11	0.5334	-0.1097	0.4519	0.062*
C12	0.57609 (13)	0.1291 (3)	0.50101 (17)	0.0524 (6)
H12	0.6124	0.0940	0.5653	0.063*
C13	0.42539 (11)	-0.0832 (3)	0.26121 (16)	0.0453 (5)
C14	0.39663 (13)	-0.0664 (4)	0.16585 (18)	0.0592 (6)
H14	0.4062	0.0328	0.1483	0.071*
C15	0.35413 (16)	-0.1948 (4)	0.0972 (2)	0.0730 (8)
H15	0.3359	-0.1824	0.0340	0.088*
C16	0.33855 (14)	-0.3410 (4)	0.1214 (2)	0.0703 (8)
H16	0.3098	-0.4273	0.0749	0.084*
C17	0.36580 (13)	-0.3588 (3)	0.2151 (2)	0.0598 (7)
H17	0.3550	-0.4569	0.2316	0.072*
C18	0.40904 (13)	-0.2318 (3)	0.28455 (19)	0.0519 (6)
H18	0.4275	-0.2457	0.3477	0.062*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0723 (2)	0.0729 (2)	0.0885 (2)	0.00301 (14)	0.0625 (2)	-0.01335 (15)
C1	0.0545 (14)	0.0446 (12)	0.0456 (13)	0.0024 (11)	0.0373 (12)	0.0014 (10)
C2	0.0533 (14)	0.0587 (15)	0.0542 (14)	-0.0045 (12)	0.0358 (13)	0.0008 (12)
C3	0.0735 (19)	0.0519 (14)	0.0538 (16)	-0.0128 (13)	0.0421 (15)	-0.0089 (12)
C4	0.085 (2)	0.0461 (14)	0.0686 (17)	0.0016 (13)	0.0585 (17)	-0.0044 (12)
C5	0.0604 (15)	0.0474 (14)	0.0635 (16)	0.0056 (12)	0.0449 (14)	0.0019 (12)
C6	0.0498 (13)	0.0417 (12)	0.0408 (12)	0.0028 (10)	0.0320 (11)	0.0049 (9)
C7	0.0478 (13)	0.0444 (12)	0.0457 (13)	0.0023 (10)	0.0338 (12)	0.0007 (10)
C8	0.0521 (14)	0.0397 (13)	0.0531 (15)	0.0063 (11)	0.0322 (13)	0.0065 (11)
C9	0.0461 (13)	0.0519 (15)	0.0438 (13)	0.0079 (11)	0.0254 (11)	0.0078 (11)
C10	0.0424 (12)	0.0472 (12)	0.0451 (12)	0.0016 (10)	0.0312 (11)	0.0007 (10)
C11	0.0570 (15)	0.0439 (13)	0.0462 (14)	0.0000 (11)	0.0323 (13)	0.0063 (11)
C12	0.0528 (14)	0.0498 (14)	0.0406 (12)	-0.0006 (11)	0.0270 (12)	0.0044 (11)
C13	0.0404 (12)	0.0480 (13)	0.0467 (13)	0.0030 (10)	0.0298 (11)	0.0010 (10)
C14	0.0581 (15)	0.0671 (17)	0.0520 (15)	-0.0102 (13)	0.0378 (13)	-0.0029 (13)
C15	0.0733 (19)	0.085 (2)	0.0504 (16)	-0.0171 (16)	0.0388 (16)	-0.0123 (15)

C16	0.0628 (18)	0.0634 (18)	0.0646 (19)	-0.0129 (14)	0.0363 (16)	-0.0165 (14)
C17	0.0541 (15)	0.0479 (14)	0.0654 (17)	-0.0005 (12)	0.0367 (14)	0.0014 (13)
C18	0.0493 (13)	0.0500 (14)	0.0504 (14)	0.0041 (11)	0.0322 (12)	0.0047 (11)

Geometric parameters (Å, °)

Br1—C1	1.897 (2)	C9—H9	0.9300
C1—C2	1.375 (3)	C10—C11	1.391 (3)
C1—C6	1.391 (3)	C10—C13	1.489 (3)
C2—C3	1.375 (4)	C11—C12	1.376 (3)
C2—H2	0.9300	C11—H11	0.9300
C3—C4	1.372 (4)	C12—H12	0.9300
C3—H3	0.9300	C13—C18	1.390 (3)
C4—C5	1.374 (4)	C13—C14	1.391 (3)
C4—H4	0.9300	C14—C15	1.378 (4)
C5—C6	1.397 (3)	C14—H14	0.9300
C5—H5	0.9300	C15—C16	1.374 (4)
C6—C7	1.489 (3)	C15—H15	0.9300
C7—C8	1.383 (3)	C16—C17	1.377 (4)
C7—C12	1.386 (3)	C16—H16	0.9300
C8—C9	1.379 (3)	C17—C18	1.380 (3)
C8—H8	0.9300	C17—H17	0.9300
C9—C10	1.389 (3)	C18—H18	0.9300
C2—C1—C6	122.4 (2)	C9—C10—C11	117.1 (2)
C2—C1—Br1	116.95 (18)	C9—C10—C13	121.9 (2)
C6—C1—Br1	120.61 (17)	C11—C10—C13	120.9 (2)
C3—C2—C1	119.5 (2)	C12—C11—C10	121.5 (2)
C3—C2—H2	120.2	C12—C11—H11	119.2
C1—C2—H2	120.2	C10—C11—H11	119.2
C4—C3—C2	119.8 (2)	C11—C12—C7	121.1 (2)
C4—C3—H3	120.1	C11—C12—H12	119.5
C2—C3—H3	120.1	C7—C12—H12	119.5
C3—C4—C5	120.4 (2)	C18—C13—C14	118.1 (2)
C3—C4—H4	119.8	C18—C13—C10	121.7 (2)
C5—C4—H4	119.8	C14—C13—C10	120.2 (2)
C4—C5—C6	121.5 (2)	C15—C14—C13	120.8 (3)
C4—C5—H5	119.2	C15—C14—H14	119.6
C6—C5—H5	119.2	C13—C14—H14	119.6
C1—C6—C5	116.3 (2)	C16—C15—C14	120.4 (3)
C1—C6—C7	123.5 (2)	C16—C15—H15	119.8
C5—C6—C7	120.2 (2)	C14—C15—H15	119.8
C8—C7—C12	117.8 (2)	C15—C16—C17	119.6 (3)
C8—C7—C6	120.5 (2)	C15—C16—H16	120.2
C12—C7—C6	121.7 (2)	C17—C16—H16	120.2
C9—C8—C7	121.2 (2)	C16—C17—C18	120.4 (3)
C9—C8—H8	119.4	C16—C17—H17	119.8
C7—C8—H8	119.4	C18—C17—H17	119.8

C8—C9—C10	121.3 (2)	C17—C18—C13	120.8 (2)
C8—C9—H9	119.3	C17—C18—H18	119.6
C10—C9—H9	119.3	C13—C18—H18	119.6
C6—C1—C2—C3	1.8 (4)	C8—C9—C10—C13	-175.5 (2)
Br1—C1—C2—C3	-179.94 (19)	C9—C10—C11—C12	-1.3 (3)
C1—C2—C3—C4	0.0 (4)	C13—C10—C11—C12	175.5 (2)
C2—C3—C4—C5	-1.4 (4)	C10—C11—C12—C7	0.2 (4)
C3—C4—C5—C6	1.0 (4)	C8—C7—C12—C11	1.0 (4)
C2—C1—C6—C5	-2.2 (3)	C6—C7—C12—C11	-178.2 (2)
Br1—C1—C6—C5	179.65 (16)	C9—C10—C13—C18	-149.7 (2)
C2—C1—C6—C7	176.8 (2)	C11—C10—C13—C18	33.6 (3)
Br1—C1—C6—C7	-1.4 (3)	C9—C10—C13—C14	31.9 (3)
C4—C5—C6—C1	0.8 (3)	C11—C10—C13—C14	-144.8 (2)
C4—C5—C6—C7	-178.2 (2)	C18—C13—C14—C15	-1.1 (4)
C1—C6—C7—C8	-113.1 (3)	C10—C13—C14—C15	177.4 (3)
C5—C6—C7—C8	65.9 (3)	C13—C14—C15—C16	1.0 (5)
C1—C6—C7—C12	66.1 (3)	C14—C15—C16—C17	-0.1 (5)
C5—C6—C7—C12	-115.0 (3)	C15—C16—C17—C18	-0.7 (4)
C12—C7—C8—C9	-0.9 (4)	C16—C17—C18—C13	0.6 (4)
C6—C7—C8—C9	178.2 (2)	C14—C13—C18—C17	0.3 (3)
C7—C8—C9—C10	-0.3 (4)	C10—C13—C18—C17	-178.2 (2)
C8—C9—C10—C11	1.4 (4)		

Hydrogen-bond geometry (Å, °)

Cg2 and Cg3 are the centroids of the C7—C12 and C13—C18 rings, respectively.

<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>
C2—H2 \cdots Cg3 ⁱ	0.93	2.84	3.778 (4)	148
C14—H14 \cdots Cg2 ⁱⁱ	0.93	2.97	3.658 (5)	147

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