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1,4-Bis(4-bromophenyl)-2,3-diphenylbenzene

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Key indicators

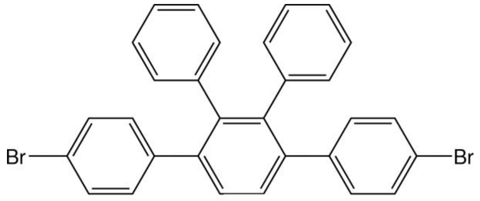
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.107
Data-to-parameter ratio = 24.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

One of our major research efforts has been the synthesis of thermally stable polymers with applications in aerospace and electronic systems. Of particular interest is the poly(p-terphenylenevinylene) polymer series and the title compound, $C_{30}H_{20}Br_2$, provides a gateway into the synthesis of functionalized polymers with an expected nonpolar organic solubility enhancement by virtue of the polyphenyl backbone.

Comment

Phenylated aromatic compounds have been used extensively in the preparation of thermally stable materials for aerospace applications, primarily because of the enhanced solubility that the pendent phenyl substituents convey (Wolfe & Arnold, 1981). Brominated derivatives in particular are useful because they can be converted to carboxylated systems (Wolfe & Arnold, 1981) or coupled directly to form rigid-rod polymers (Naarmann & Kallitsis, 1992; Kallitsis & Naarmann, 1991). In this context, the crystal structure of the title compound, (I), has been determined (Fig. 1).



(I)

The crystal packing of (I) is very interesting. The orientations of the benzene rings (Fig. 2 and Table 1) allow for very compact layering in the crystal structure, held together by a network of C—H$\cdots\pi$ attractions.

The packing shows that the C1–C6, C11–C16 and C41–C46 benzene rings have parallel orientations relative to the same rings on a neighboring molecule. The same is true for the C21–C26 and C31–C35 rings. This suggests that the potential energy of interaction between neighboring molecules is minimized by allowing π–π interactions between benzene rings, and the packing is more efficient when the brominated rings are oriented in a similar manner and when the non-brominated rings are also oriented similarly (Fig. 3). Upon close inspection of the structure, one can see that the molecule is slightly bent about its center; the C1–C6 ring is not entirely planar. The angle between the plane defined by C1, C2 and C3 and that of C4, C5 and C6 of the central ring is 2.5 (3)°. This is due to the strong coulombic repulsive forces from both the bromine atoms and benzene rings on adjacent molecules. Due to the packing, these atoms are placed in very close proximity to one

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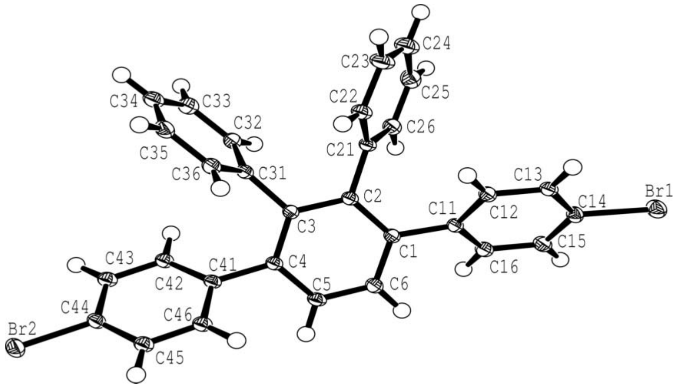

Figure 1
The molecular structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids at the 40% probability level.

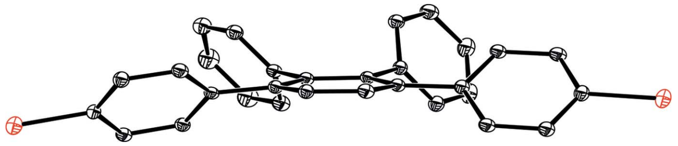

Figure 2
View of (I), showing the orientation of the central and peripheral benzene rings. H atoms have been omitted.

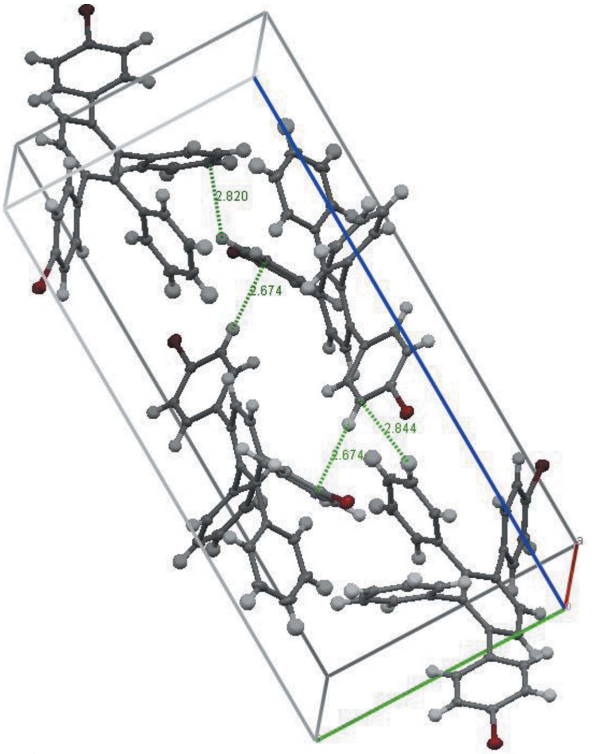

Figure 3
The unit cell contents for (I), showing the C-H$\cdots\pi$ contacts (dashed lines).

another. The bromine atoms, having a much larger van der Waals radius than carbon, repel each other even at a distance where repulsions between the benzene rings is not an issue. Also, due to the benzene rings being interwoven with adjacent molecules to maximize π–π interactions, the loss of intramolecular mobility may contribute to the non-planarity of the central ring. Angles between the various benzene rings in the molecule are listed in Table 1.

Bond distances and angles within the structure are within expected ranges (Cambridge Structural Database, 2005 Version; Allen, 2002).

Experimental

A solution of 5.00 g (9.22 mmol) of 2,5-bis(4-bromophenyl)-3,4-diphenylcyclopentadiene (Coan *et al.*, 1955) and 3.40 g (37 mmol) of bicyclo[2.2.1]hepta-2,5-diene in 20 ml of toluene was refluxed (383 K) for 16 h. After the intense purple solution had turned light pink, it was cooled and the precipitate was filtered and washed with 20 ml of methanol, yielding 3.49 g (70%) of an off-white product (m.p. 552.3–555.5 K; literature 548–551 K (Wolfe & Arnold, 1981; Arnold, 1977). Crystals suitable for X-ray crystallographic analysis were obtained from CH_2Cl_2.

Crystal data

$C_{30}H_{20}Br_2$
$M_r = 540.28$
Monoclinic, $P2_1/n$
$a = 10.4922$ (6) Å
$b = 9.9679$ (5) Å
$c = 22.4589$ (12) Å
$\beta = 96.5800$ (14)°
$V = 2333.4$ (2) Å^3
$Z = 4$
$D_x = 1.538$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 3.49$ mm^{-1}
$T = 173$ (2) K
Block, colorless
0.41 × 0.28 × 0.25 mm

Data collection

Bruker AXS SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2003)
$T_{\min} = 0.345$, $T_{\max} = 0.418$
26710 measured reflections
7124 independent reflections
6306 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 30.6°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.107$
$S = 1.18$
7124 reflections
289 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 3P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.93$ e Å^{-3}
$\Delta\rho_{\min} = -0.52$ e Å^{-3}

Table 1
Table 1. Angles between least-squares planes (°) for the rings in (I). For each ring, angles are given for the subsequent rings in the list.

Atoms	angles			
C1–C6	47.68 (10)	59.33 (11)	66.42 (11)	48.08 (10)
C11–C16		58.09 (11)	75.22 (11)	85.92 (11)
C21–C26			59.72 (12)	88.13 (12)
C31–C36				51.11 (12)
C41–C46				

H atoms were positioned geometrically (C—H = 0.96 Å) and refined as riding, with $U_{\rm iso}(H) = 1.3U_{\rm eq}(C)$.

Data collection: *SMART* (Bruker, 1997–2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *OSCAIL* (McArdle, 1995) and *PLATON* (Spek, 2003).

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