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2,2′-Dibromo-3,3′,4,4′,5,5′,6,6′-octamethyl-1,1′-biphenyl

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Key indicators

Single-crystal X-ray study
T = 174 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.038
wR factor = 0.050
Data-to-parameter ratio = 10.5

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

The title compound, $C_{29}H_{24}Br_2$, has crystallographic twofold rotation symmetry. The Br atom in the 2 position is disordered with the methyl group in the 6 position. The biphenyl bridge bond distance and the torsion angle between the rings are in good agreement with similar previously reported structures.

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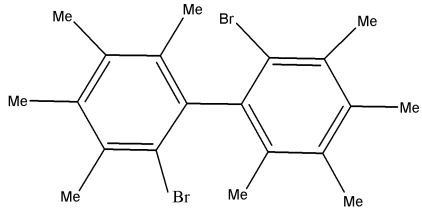
Comment

The title compound, (I), crystallizes in the space group *Pbcn*, with the molecule on a crystallographic twofold axis, which bisects the angle between the two rings. One half of the molecule consists of a benzene ring, to which are bonded a Br atom and four methyl groups. The Br atom is bonded in the 2 position and is disordered between the 2 and 6 positions. The methyl group in the 6 position is concomitantly disordered.



(I)

The major-component C2—Br1 distance is 1.871 (4) Å and the minor-component C6—Br2 distance is 1.809 (5) Å. The major-component C2—Br1 distance is comparable to the C—Br distances in 2,2′-dibromobiphenyl (1.895 and 1.905 Å; MacNeil & Decken, 1999).

The C—C distance of the biphenyl bridge is 1.502 (9) Å, similar to that of 2,2′-dibromobiphenyl (1.499 Å), decachlorobiphenyl (1.522 Å; Pedersen, 1975) and decakis(dichloromethyl)biphenyl (1.534 Å; Biali *et al.*, 1988).

The interplanar angle between the phenyl rings in the molecule is 85.2 (8)°. This compares well with the corresponding angles in 2,2′-dibromobiphenyl (86.0°), decachlorobiphenyl (86.7°) and decakis(dichloromethyl)biphenyl (84.4°).

Experimental

The title compound was synthesized according to a previously reported method (Shibata *et al.*, 2003).

Crystal data

$C_{20}H_{24}Br_2$
M_r = 424.22
Orthorhombic, *Pbcn*
a = 13.530 (4) Å
b = 10.987 (4) Å
c = 11.916 (3) Å
V = 1771.4 (9) Å^3
Z = 4
D_x = 1.591 Mg m^{-3}
Mo Kα radiation
Cell parameters from 2260 reflections
θ = 2.4–24.0°
μ = 4.59 mm^{-1}
T = 174.2 K
Block, colorless
0.29 × 0.21 × 0.13 mm

Data collection

Bruker SMART 1000 diffractometer
ω scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.280, T_{max} = 0.554
7776 measured reflections
1483 independent reflections
1061 reflections with $I > 3\sigma(I)$
R_{int} = 0.046
θ_{max} = 24.7°
$h = -13 \rightarrow 15$
$k = -12 \rightarrow 12$
$l = -13 \rightarrow 13$

Refinement

Refinement on F
R = 0.038
wR = 0.050
S = 1.85
1061 reflections
101 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o) + 0.00022|F_o|^2]$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C2	1.871 (4)	C1—C1^{i}	1.502 (9)
Br2—C6	1.809 (5)		
C2—C1—C1^{i}—C2^{i}	−83.7 (8)	C2—C1—C1^{i}—C6^{i}	95.6 (3)

Symmetry code: (i) $1-x, y, \frac{3}{2}-z$.

Table 2
Contact distances (Å).

Br1···Br2^{ii}	3.557 (2)	Br2···Br2^{iv}	3.532 (4)
Br2···C11^{iii}	3.502 (2)		

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

Atoms C10 and C11 were inserted along the C—Br bonds at a fixed distance of 1.54 Å from the ring C atoms. The displacement parameters of these atoms were fixed at 0.0456 Å^2 because their positions are not well defined and the C—Br distance is too short to allow for proper resolution of the disordered atoms. The Br1 atom occupancy was allowed to refine and then fixed at 0.73. The occupancies of atoms Br2, C10 and C11 were tied appropriately to that of Br1.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation & Rigaku, 1998); molecular graphics: *TEXSAN*; software used to prepare material for publication: *TEXSAN*.

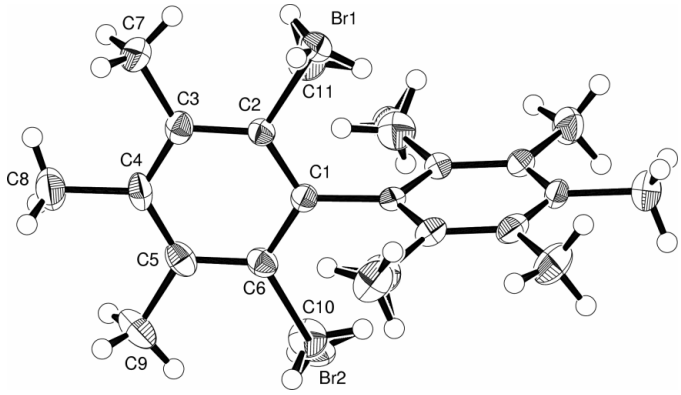


Figure 1
The molecular structure of the title compound, including the disorder. Displacement ellipsoids are drawn at the 50% probability level.

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References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Biali, S. E., Kahr, B., Okamoto, Y., Aburatani, R. & Mislow, K. (1988). *J. Am. Chem. Soc.* **110**, 1917–1922.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Bruker (2003). *SMART* (Version 5.631) and *SAINT* (Version 6.40). Bruker AXS Inc., Madison, Wisconsin, USA.
MacNeil, D. D. & Decken, A. (1999). *Acta Cryst.* C**55**, 628–630.
Molecular Structure Corporation & Rigaku (1998). *TEXSAN*. Version 1.10. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA, and Rigaku Corporation, 3-9-12 Akishima, Tokyo, Japan.
Pedersen, B. F. (1975). *Acta Cryst.* B**31**, 2931–2933.
Shibata, T., Tsuruta, H., Danjo, H. & Imamoto, T. (2003). *J. Mol. Cat. A*, **196**, 117–124.