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4,4′-Bibenzocyclobutene

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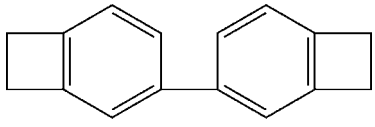
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.050; wR factor = 0.140; data-to-parameter ratio = 15.5.

In the molecule of the title compound, $C_{16}H_{14}$, the dihedral angles between the planar rings are 1.92 (3) and 0.27 (3)° between the two pairs of fused rings, and 38.00 (4)° between the two six-membered rings.

Related literature

For general background, see: Allen *et al.* (1987); Kirchhoff & Bruza (1993); Corley & Wong (1992).



Experimental

Crystal data

$C_{16}H_{14}$
M_r = 206.27
Monoclinic, $P2_1/n$
a = 8.0250 (16) Å
b = 11.365 (2) Å
c = 12.850 (3) Å
β = 101.19 (3)°
V = 1149.7 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 298 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.980, T_{max} = 0.987
2248 measured reflections
2248 independent reflections
1414 reflections with $I > 2\sigma(I)$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.140
S = 1.08
2248 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2237).

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supplementary materials

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4,4'-Bibenzocyclobutene

Z.-Q. Feng, H. Cang, W.-W. Tian, S.-L. Dong and J.-T. Wang

Comment

Benzocyclobutenes (BCBs) are a family of thermally polymerisable monomers for high-performance polymers, which have been widely used in the microelectronics industry because of their low dielectric constant, low dispersion factor, low water up-take, high thermal and chemical stabilities, and ease of processing (Kirchhoff & Bruza, 1993). Homopolymers and copolymers of aryl bridged bisbenzocyclobutene monomers (bisBCB) also exhibit excellent properties (Corley & Wong, 1992).

In the molecule of the title compound, (I), (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). Rings A (C1—C3/C8), B (C3—C8), C (C9—C14) and D (C11/C12/C15/C16) are, of course, planar and the dihedral angles between them are A/B = 1.92 (3)°, C/D = 0.27 (3)° and B/C = 38.00 (4)°.

As can be seen from the packing diagram, (Fig. 2), the molecules are elongated along the c axis and stacked along the b axis.

Experimental

A mixture of benzocyclobutene-4-boronic acid (29.6 mg, 0.20 mmol), 4-bromobenzocyclobutene (85.0 mg, 0.46 mmol), tetrabutylammonium bromide (6.44 mg, 0.02 mmol) and palladium chloride (2.83 mg, 0.016 mmol) in ethanol (10 ml) was stirred for 1 h under nitrogen atmosphere at room temperature. Potassium carbonate (580 mg, 0.42 mmol) was then added and the mixture was stirred vigorously for 22 h. The reaction mixture was poured into distilled water (15 ml) and then extracted with chloroform (20 ml) three times. The combined organic phase was washed with distilled water (30 ml) and dried over sodium sulfate. Removal of the solvent under reduced pressure gave a grey-white solid, which could be purified by chromatography on silica gel using petroleum ether, as eluent to recover excess 4-bromobenzocyclobutene first, then using a mixture of petroleum ether and toluene (25:1) to afford (I) (yield; 37.0 mg, 90%, m.p. 333 K). Crystals of (I) suitable for X-ray diffraction was obtained by slow evaporation of hexane.

Refinement

H atoms were positioned geometrically, with C—H = 0.93 and 0.97 Å for aromatic and methylene H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

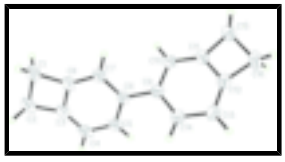

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Fig. 2. A packing diagram for (I).

4,4'-bibenzocyclobutene

Crystal data

$C_{16}H_{14}$	F_{000} = 440
M_r = 206.27	D_x = 1.192 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 333 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.0250 (16) Å	Cell parameters from 25 reflections
b = 11.365 (2) Å	θ = 10–13º
c = 12.850 (3) Å	μ = 0.07 mm^{-1}
β = 101.19 (3)º	T = 298 (2) K
V = 1149.7 (4) Å^3	Block, colorless
Z = 4	0.30 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.0000
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 2.4º
T = 298(2) K	h = −9→9
ω/2θ scans	k = 0→14
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→15
T_{min} = 0.980, T_{max} = 0.987	3 standard reflections
2248 measured reflections	every 200 reflections
2248 independent reflections	intensity decay: none
1414 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0408P)^2 + 0.3817P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.140	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
S = 1.08	$\Delta\rho_{min}$ = −0.13 e Å^{-3}
2248 reflections	Extinction correction: none
145 parameters	
Primary atom site location: structure-invariant direct methods	

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.0790 (4)	0.9190 (3)	0.31338 (18)	0.0752 (8)
H1A	0.1490	0.9893	0.3212	0.090*
H1B	−0.0403	0.9381	0.2901	0.090*
C2	0.1442 (3)	0.8183 (3)	0.24864 (19)	0.0748 (8)
H2A	0.0580	0.7859	0.1925	0.090*
H2B	0.2470	0.8375	0.2231	0.090*
C3	0.1737 (3)	0.7478 (2)	0.34986 (18)	0.0589 (6)
C4	0.2271 (3)	0.6419 (2)	0.3941 (2)	0.0673 (7)
H4A	0.2675	0.5831	0.3553	0.081*
C5	0.2181 (3)	0.6263 (2)	0.50072 (19)	0.0600 (6)
H5A	0.2547	0.5555	0.5336	0.072*
C6	0.1557 (3)	0.7139 (2)	0.55964 (17)	0.0494 (6)
C7	0.1062 (3)	0.8223 (2)	0.51261 (17)	0.0552 (6)
H7A	0.0685	0.8829	0.5508	0.066*
C8	0.1155 (3)	0.8361 (2)	0.40726 (17)	0.0558 (6)
C9	0.1413 (3)	0.6890 (2)	0.67190 (17)	0.0502 (6)
C10	0.1747 (3)	0.7754 (2)	0.74977 (17)	0.0531 (6)
H10A	0.2084	0.8507	0.7344	0.064*
C11	0.1560 (3)	0.7448 (2)	0.85081 (17)	0.0518 (6)
C12	0.1065 (3)	0.6355 (2)	0.87572 (18)	0.0559 (6)
C13	0.0705 (3)	0.5482 (2)	0.8009 (2)	0.0624 (7)
H13A	0.0351	0.4738	0.8177	0.075*
C14	0.0897 (3)	0.5770 (2)	0.69819 (18)	0.0561 (6)
H14A	0.0675	0.5198	0.6455	0.067*
C15	0.1723 (3)	0.7919 (2)	0.96359 (17)	0.0634 (7)
H15A	0.0934	0.8546	0.9710	0.076*
H15B	0.2874	0.8116	0.9981	0.076*
C16	0.1122 (4)	0.6654 (2)	0.99105 (19)	0.0743 (8)
H16A	0.1963	0.6215	1.0405	0.089*
H16B	0.0023	0.6640	1.0119	0.089*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.091 (2)	0.085 (2)	0.0501 (14)	−0.0114 (16)	0.0156 (14)	0.0022 (14)
C2	0.0746 (18)	0.104 (2)	0.0470 (14)	−0.0247 (16)	0.0143 (13)	−0.0124 (14)
C3	0.0588 (15)	0.0726 (17)	0.0474 (13)	−0.0176 (13)	0.0153 (11)	−0.0137 (12)
C4	0.0708 (17)	0.0742 (18)	0.0616 (16)	−0.0081 (14)	0.0244 (13)	−0.0249 (14)
C5	0.0663 (16)	0.0543 (15)	0.0627 (15)	−0.0014 (12)	0.0207 (12)	−0.0090 (12)
C6	0.0493 (13)	0.0538 (14)	0.0466 (12)	−0.0040 (11)	0.0133 (10)	−0.0033 (11)
C7	0.0649 (15)	0.0566 (14)	0.0475 (13)	−0.0039 (12)	0.0190 (11)	−0.0045 (11)
C8	0.0562 (14)	0.0659 (16)	0.0456 (13)	−0.0089 (12)	0.0103 (11)	−0.0033 (12)
C9	0.0493 (13)	0.0526 (14)	0.0509 (13)	−0.0027 (11)	0.0150 (10)	−0.0007 (11)
C10	0.0583 (14)	0.0491 (13)	0.0538 (14)	−0.0041 (11)	0.0154 (11)	−0.0001 (11)
C11	0.0493 (13)	0.0601 (15)	0.0465 (13)	0.0039 (11)	0.0106 (10)	0.0008 (11)
C12	0.0541 (14)	0.0611 (15)	0.0537 (14)	0.0060 (12)	0.0133 (11)	0.0095 (12)
C13	0.0701 (17)	0.0514 (15)	0.0701 (16)	−0.0004 (12)	0.0243 (13)	0.0117 (13)
C14	0.0597 (15)	0.0515 (14)	0.0588 (14)	−0.0037 (12)	0.0159 (12)	−0.0037 (12)
C15	0.0622 (16)	0.0784 (18)	0.0483 (13)	0.0037 (14)	0.0073 (11)	−0.0027 (13)
C16	0.0791 (19)	0.093 (2)	0.0537 (15)	0.0096 (16)	0.0203 (13)	0.0148 (14)

Geometric parameters (Å, °)

C1—C8	1.514 (3)	C9—C10	1.390 (3)
C1—C2	1.564 (4)	C9—C14	1.401 (3)
C1—H1A	0.9700	C10—C11	1.380 (3)
C1—H1B	0.9700	C10—H10A	0.9300
C2—C3	1.507 (3)	C11—C12	1.361 (3)
C2—H2A	0.9700	C11—C15	1.526 (3)
C2—H2B	0.9700	C12—C13	1.373 (3)
C3—C4	1.364 (4)	C12—C16	1.512 (3)
C3—C8	1.380 (3)	C13—C14	1.397 (3)
C4—C5	1.397 (3)	C13—H13A	0.9300
C4—H4A	0.9300	C14—H14A	0.9300
C5—C6	1.401 (3)	C15—C16	1.578 (4)
C5—H5A	0.9300	C15—H15A	0.9700
C6—C7	1.395 (3)	C15—H15B	0.9700
C6—C9	1.497 (3)	C16—H16A	0.9700
C7—C8	1.379 (3)	C16—H16B	0.9700
C7—H7A	0.9300		
C8—C1—C2	86.3 (2)	C10—C9—C14	119.6 (2)
C8—C1—H1A	114.3	C10—C9—C6	121.3 (2)
C2—C1—H1A	114.3	C14—C9—C6	119.1 (2)
C8—C1—H1B	114.3	C11—C10—C9	117.2 (2)
C2—C1—H1B	114.3	C11—C10—H10A	121.4
H1A—C1—H1B	111.4	C9—C10—H10A	121.4
C3—C2—C1	86.72 (18)	C12—C11—C10	122.8 (2)
C3—C2—H2A	114.2	C12—C11—C15	94.11 (19)

C1—C2—H2A	114.2	C10—C11—C15	143.1 (2)
C3—C2—H2B	114.2	C11—C12—C13	121.8 (2)
C1—C2—H2B	114.2	C11—C12—C16	94.1 (2)
H2A—C2—H2B	111.4	C13—C12—C16	144.1 (2)
C4—C3—C8	122.0 (2)	C12—C13—C14	116.5 (2)
C4—C3—C2	144.4 (2)	C12—C13—H13A	121.8
C8—C3—C2	93.5 (2)	C14—C13—H13A	121.8
C3—C4—C5	116.7 (2)	C13—C14—C9	122.2 (2)
C3—C4—H4A	121.6	C13—C14—H14A	118.9
C5—C4—H4A	121.6	C9—C14—H14A	118.9
C4—C5—C6	122.1 (2)	C11—C15—C16	85.38 (19)
C4—C5—H5A	119.0	C11—C15—H15A	114.4
C6—C5—H5A	119.0	C16—C15—H15A	114.4
C7—C6—C5	119.7 (2)	C11—C15—H15B	114.4
C7—C6—C9	121.0 (2)	C16—C15—H15B	114.4
C5—C6—C9	119.3 (2)	H15A—C15—H15B	111.5
C8—C7—C6	117.4 (2)	C12—C16—C15	86.41 (18)
C8—C7—H7A	121.3	C12—C16—H16A	114.3
C6—C7—H7A	121.3	C15—C16—H16A	114.3
C7—C8—C3	122.0 (2)	C12—C16—H16B	114.3
C7—C8—C1	144.5 (2)	C15—C16—H16B	114.3
C3—C8—C1	93.4 (2)	H16A—C16—H16B	111.4
C8—C1—C2—C3	−0.28 (19)	C7—C6—C9—C14	141.2 (2)
C1—C2—C3—C4	178.6 (4)	C5—C6—C9—C14	−38.0 (3)
C1—C2—C3—C8	0.3 (2)	C14—C9—C10—C11	0.5 (3)
C8—C3—C4—C5	0.8 (4)	C6—C9—C10—C11	179.2 (2)
C2—C3—C4—C5	−177.2 (3)	C9—C10—C11—C12	−0.2 (3)
C3—C4—C5—C6	0.7 (4)	C9—C10—C11—C15	179.1 (3)
C4—C5—C6—C7	−2.4 (4)	C10—C11—C12—C13	−0.6 (4)
C4—C5—C6—C9	176.9 (2)	C15—C11—C12—C13	179.9 (2)
C5—C6—C7—C8	2.5 (3)	C10—C11—C12—C16	−179.6 (2)
C9—C6—C7—C8	−176.8 (2)	C15—C11—C12—C16	0.8 (2)
C6—C7—C8—C3	−1.0 (4)	C11—C12—C13—C14	0.9 (4)
C6—C7—C8—C1	176.4 (3)	C16—C12—C13—C14	179.3 (3)
C4—C3—C8—C7	−0.6 (4)	C12—C13—C14—C9	−0.6 (4)
C2—C3—C8—C7	178.2 (2)	C10—C9—C14—C13	−0.1 (4)
C4—C3—C8—C1	−179.1 (2)	C6—C9—C14—C13	−178.8 (2)
C2—C3—C8—C1	−0.3 (2)	C12—C11—C15—C16	−0.8 (2)
C2—C1—C8—C7	−177.5 (3)	C10—C11—C15—C16	179.8 (3)
C2—C1—C8—C3	0.3 (2)	C11—C12—C16—C15	−0.8 (2)
C7—C6—C9—C10	−37.5 (3)	C13—C12—C16—C15	−179.4 (4)
C5—C6—C9—C10	143.3 (2)	C11—C15—C16—C12	0.73 (18)

Fig. 1

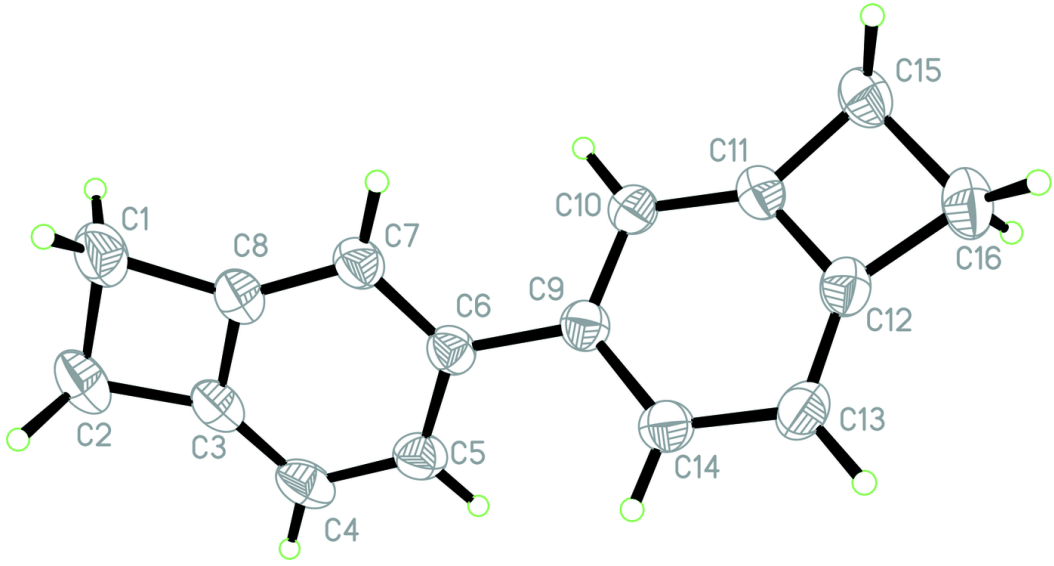

Fig. 2