was prepared using *ORTEP* (Johnson, 1965) incorporated in *PLATON* and Fig. 2 was prepared using *PLUTON* (Spek, 1991). Material was prepared for publication using *PLATON*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1146). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5,10-Diiodo-5,10-dihydroboranthrene

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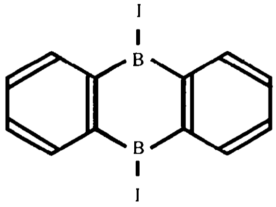
Abstract

The structure of the title compound, 9,10-diiodo-9,10-dihydro-9,10-diboranthracene, $C_{12}H_8B_2I_2$, has been determined. The nearly planar skeleton of the molecule suggests that the central six-membered boron-containing ring is conjugated with the outer two benzene rings. The molecules stack along the *c* axis and the B atoms of one molecule lie just above the I atoms of the next molecule.

Comment

As part of our study of organic donor–acceptor molecular complexes, the title compound was interesting because it was possible that it possessed an electron-accepting property. Only four kinds of 5,10-disubstituted-5,10-dihydroboranthrene compound have been synthesized, namely, the diiodo derivative (Siebert, Schaper & Asgarouladi, 1974), the dibromo derivative (Asgarouladi, Full, Schaper & Siebert, 1974), the dichloro derivative (Clement, 1965) and the dimethyl derivative (Schacht & Kaufmann, 1987). However, none of these compounds has so far been structurally analyzed. This report is concerned with the diiodo derivative, (I).



(I)

Since there are two molecules of (I) in the unit cell of space group *C*2/*m*, a quarter of the molecule corresponds to an asymmetric unit. The symmetry of the molecule is C_{2h} and is very close to D_{2h}. The mirror plane passes through the I—B···B—I atoms perpendicular to the molecular plane and a twofold axis, perpendicular to the mirror, is present on the molecular plane. All the atoms of the molecule lie on a plane, within experimental error, except for the I atoms. The angle between the least-squares molecular plane and the I—B bond is 175.6°. Though the concentrated electron density at the I atoms (about 54% in the whole molecule) results in an average bond-length e.s.d. as large as 0.01 Å, the C(1)—C(2), C(2)—C(3) and C(3)—C(3^i) (symmetry codes are in Table 2) bond lengths are in the conjugated C_{sp^2}—C_{sp^2} range. A slightly large value for the C(1)—C(1^i) bond length, however, suggests that the π-bond character of this bond is weakened. In addition, the B—C(1) bond length of 1.52 (1) Å is significantly shorter than the distance of 1.578 (1) Å found in $B(CH_3)_3$ (Bartell & Carroll, 1965) and the distances 1.571 (3)–1.589 (5) Å found in $B(Ph)_3$ (Zettler, Hausen & Hess, 1974). These facts imply that the π electrons of the outer benzene rings are partly donated to the empty 2*p* orbitals of the B atoms and that the conjugated system is enlarged to include the whole molecule. The I—B bond length of 2.18 (1) Å is in the range of ordinary covalent bonds (Cotton & Wilkinson, 1980), suggesting that electron donation does not occur from the I to the B atom.

All the molecules stack along the *c* axis, with their molecular planes parallel to each other, as shown in Fig. 2, and at an angle of 58.5° to the *c* axis. The interplanar distance between the least-squares molecular planes is 3.63 Å. Viewed perpendicular to the molecular plane, a B atom of one molecule is located just above an I atom of the next molecule.

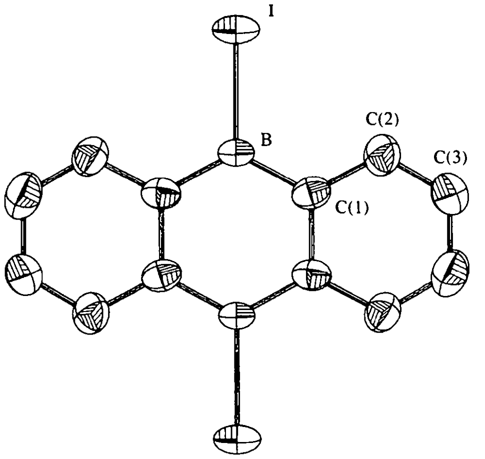


Fig. 1. The molecular structure of the title compound with the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level (*ORTEP*II; Johnson, 1976) and the H atoms are omitted.

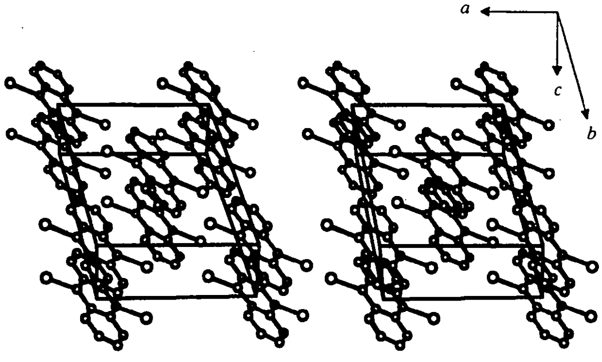


Fig. 2. A stereoscopic molecular packing view of (I) in the unit cell.

Experimental

The title compound was obtained from the reaction of triiodoborane with 1,2-diiodobenzene according to the literature method of Siebert, Schaper & Asgarouladi (1974). Crystals were grown by sublimation *in vacuo* in the course of purification.

Crystal data

$C_{12}H_8B_2I_2$
M_r = 427.63
Monoclinic
$C2/m$
a = 10.246 (2) Å
b = 14.487 (2) Å
c = 4.2536 (5) Å
β = 90.23 (1)°
V = 631.4 (2) Å^3
Z = 2
D_x = 2.250 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 20 reflections
θ = 14.5–15.6°
μ = 5.01 mm^{-1}
T = 293 K
Prism
0.4 × 0.2 × 0.2 mm
Yellow

Data collection

Rigaku AFC-5 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1072 measured reflections
957 independent reflections
792 observed reflections [$F > 3\sigma(F)$]

$R_{\rm int}$ = 0.01
$\theta_{\rm max}$ = 30°
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 5$
3 standard reflections monitored every 100 reflections intensity decay: 7%

Refinement

Refinement on F
R = 0.054
wR = 0.039
S = 1.53
790 reflections
46 parameters
Only coordinates of H atoms refined

$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\rm max}$ = 0.23
$\Delta\rho_{\rm max}$ = 1.9 e Å^{-3}
$\Delta\rho_{\rm min}$ = −1.8 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/6\pi^2)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
I	0.69029 (7)	0	0.4023 (2)	0.0577 (5)
C(1)	0.9395 (6)	0.0924 (5)	0.088 (2)	0.041 (4)
C(2)	0.8854 (7)	0.1796 (6)	0.172 (2)	0.052 (5)
C(3)	0.9420 (7)	0.2621 (5)	0.086 (2)	0.061 (6)
B	0.879 (1)	0	0.170 (3)	0.037 (7)

Table 2. *Selected geometric parameters (Å, °)*

I—B	2.18 (1)	C(1)—C(1$^{\rm i}$)	1.45 (1)
B—C(1)	1.52 (1)	C(2)—C(3)	1.38 (1)
C(1)—C(2)	1.43 (1)	C(3)—C(3$^{\rm i}$)	1.40 (2)
I—B—C(1)	117.9 (7)	C(1$^{\rm i}$)—C(1)—C(2)	117.6 (7)
C(1)—B—C(1$^{\rm ii}$)	124.2 (9)	C(1)—C(2)—C(3)	122.5 (8)
C(2)—C(1)—B	124.5 (7)	C(2)—C(3)—C(3$^{\rm i}$)	119.9 (9)
C(1$^{\rm i}$)—C(1)—B	117.9 (7)		

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

Though the crystal used for the X-ray analysis was sealed in a glass capillary because of its water sensitive nature, the intensity decay amounted to 7% in F. An absorption correction was not applied because of the instability of the specimen. The structure was solved by direct methods using *MULTAN*84 (Main, Germain & Woolfson, 1984). H atoms were located from a difference map and refined isotropically; B = 4.0 Å^2. Block-diagonal least-squares refinement was carried out with the Rigaku RASA-5*P* program package, followed by full-matrix least-squares refinement with the *UNICS* program system (Sakurai, 1967). In the final cycle, two reflections (020 and 040) were given zero weight, being seriously affected by extinction. In the final difference map, $\Delta\rho$ excursions of 1.9 and −1.8 e Å^{-3} were found near the I atoms owing to the truncation effect. Computations were performed using Panafacom U-1200II and HITAC M-680H computers at the Computer Centre of the University of Tokyo, Japan.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1194). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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cis-1,5-Dimethyl-2,4-dinitro-2,4-diazabicyclo[3.2.0]heptan-3-one and *cis*-1,5-Dimethyl-2,4-dinitro-2,4-diazabicyclo[3.1.0]hexan-3-one

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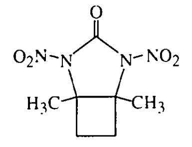
Abstract

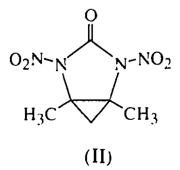
The title compounds, $C_7H_{10}N_4O_5$, (I), and $C_6H_8N_4O_5$, (II), each consist of a five-membered ring fused to a smaller ring. In compound (I), the C—C bridging bond distance [1.571 (4) Å] is longer than the expected distance of 1.54 Å for a C—C bond in a cyclobutane ring. In compound (II), however, the corresponding C—C bridging bond distance [1.507 (5) Å] is consistent with the expected bond length (1.51 Å) in a cyclopropane ring. The increase in the C—C bridge distance in compound (I) is most likely due to steric effects resulting from the additional methyl substituents.

Comment

The ideal bond distances for cyclobutane and cyclopropane rings are 1.54 and 1.51 Å, respectively (Wilson, 1992). A search of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) revealed 14 structures with the same bicyclic ring system as that found in compound (I) and 31 structures with the same bicyclic ring system as that found in compound (II).



(I)



(II)

In all but two of the structures similar to compound (I), the zero-bridged bond was longer than the expected value of 1.54 Å [the mean bond length was 1.558 (4) Å and the range of observed values was 1.525–1.583 Å]. There is, however, no correlation of this bond length with the electronegativity of the substituents on the bridged C atoms. In compound (I), the bridge distance (*i.e.* C1—C5) is 1.571 (4) Å (Fig. 1). Here, and in the related compounds, the bond length increase is most likely due to steric effects. In the opposing cyclobutane C—C distance involving unbridged C atoms, where strain due to crowding is less likely, the electronegativity of the substituents is well correlated with the bond distance.

In compound (II), the C1—C5 bond distance is 1.507 (5) Å, which is consistent with the expected value (Fig. 2). In the 31 similar structures found in the Cambridge Structural Database, the mean C—C bridge distance was 1.513 (4) Å and the range 1.464–1.561 Å.

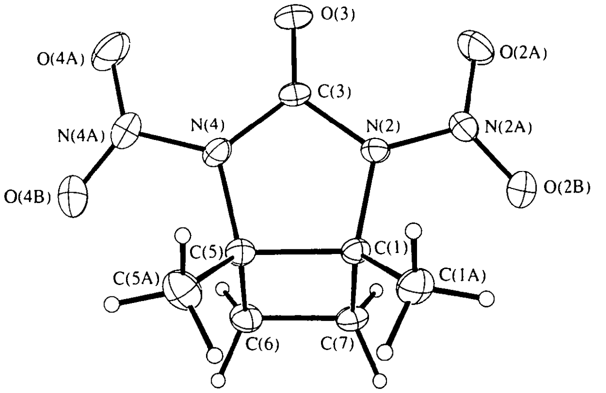


Fig. 1. View of compound (I) showing the labeling of the non-H atoms. Displacement ellipsoids are shown at 20% probability levels and H atoms are drawn as small circles of arbitrary radii.