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## Structure of 8-[1,2-Dicarba-*closo*-dodecaboran(12)-3-yl]-5,6-dicarba-*nido*-dodecaborane(12)

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**Abstract.** *iso*-C<sub>4</sub>H<sub>22</sub>B<sub>18</sub>, *M<sub>r</sub>* = 265.02, monoclinic, *C*2/*c*, *Z* = 8, *a* = 21.099 (9), *b* = 7.081 (2), *c* = 22.065 (9) Å, β = 94.27 (3)°, *U* = 3287.4 Å<sup>3</sup>, *D<sub>x</sub>* = 1.071 Mg m<sup>-3</sup>, μ(Cu Kα) = 0.254 mm<sup>-1</sup>. The structure was solved by direct methods and refined to *R* = 0.054 for 2108 counter reflections. The molecule represents the first-known case of an uncharged combination of a *nido*- and *closo*-carbaborane.

**Introduction.** Two isomeric carbaboranes, *neo*-C<sub>4</sub>H<sub>22</sub>B<sub>18</sub> and *iso*-C<sub>4</sub>H<sub>22</sub>B<sub>18</sub>, were isolated in the oxidation of the 7,8-C<sub>2</sub>H<sub>12</sub>B<sub>9</sub><sup>-</sup> anion with chromic acid (Janoušek, 1973; Plešek & Heřmánek, 1974). The molecule of the former isomer consists of two identical, mutually coupled *nido*-7,8-C<sub>2</sub>H<sub>11</sub>B<sub>9</sub> units (Janoušek, Heřmánek, Plešek & Štibr, 1974; Sklenář & Hašek, 1978).

For the constitution of the second *iso*-C<sub>4</sub>H<sub>22</sub>B<sub>18</sub> isomer a linkage of two different moieties, 1,2-C<sub>2</sub>H<sub>11</sub>B<sub>10</sub> and 5,6-C<sub>2</sub>H<sub>11</sub>B<sub>8</sub>, was proposed from the mass and NMR spectral data (Janoušek, Plešek, Štibr & Heřmánek, 1982). The X-ray study on *iso*-C<sub>4</sub>H<sub>22</sub>B<sub>18</sub> has been undertaken in order to clarify the way in which these two dicarbaborane units are connected and to determine the geometry of the molecule. The distances and angles in the 5,6-C<sub>2</sub>H<sub>11</sub>B<sub>8</sub> part of the molecule are also of great interest because unsubstituted 5,6-C<sub>2</sub>H<sub>12</sub>B<sub>8</sub> has proved to be the important readily available compound from which many new derivatives have been prepared (Štibr, Plešek

& Heřmánek, 1974; Janoušek, Plešek & Plzák, 1979; Jung & Hawthorne, 1980; Colquhoun, Greenhough & Wallbridge, 1980).

A crystal 0.35 × 0.40 × 0.60 mm was used for data collection on a Syntex *P*<sub>21</sub> diffractometer with graphite-monochromated Cu Kα radiation. The cell parameters were determined by least squares from 25 high-order reflections. Systematic extinctions indicated space group *Cc* or *C*2/*c*; the latter has been confirmed by a statistical test. The intensities of 2244 independent reflections (θ ≤ 57°) were measured in the ω/2θ scan mode, of which 2108 with *I* > 1.96σ(*I*) were considered as observed and included in further calculations. Reflections were corrected for Lorentz and polarization effects but not for absorption or extinction.

The structure was solved with *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974). For the solution the known configuration of the 12 atoms in the icosahedral carbaborane cage was used (Šubrtová, Linek & Hašek, 1980), with random position and orientation. The *E* map showed the positions of all non-H atoms. The refinement was carried out by block-diagonal least squares with a modified *NRC*-10 program (Ahmed, Hall, Pippy & Huber, 1966). The function minimized was  $w(|F_o| - |F_c|)^2$ , with  $w^{-1} = \sigma^2(F) + (0.022F_o)^2$ . A difference map showed all H atoms including two in the hydrogen bridges. Least-squares calculations, with isotropic thermal parameters for H and anisotropic parameters for the other atoms, reduced *R* ( $\sum ||F_c| - |F_o|| / \sum |F_o|$ ) to a final

value of 0.054. The final positional and isotropic thermal parameters are given in Tables 1 and 2.\*

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38045 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates ( $\times 10^4$ ) with e.s.d.'s in parentheses and  $B_{eq}$  values (Hamilton, 1959) for non-H atoms

	x	y	z	$B_{eq}$ (Å <sup>2</sup> )
C(1)	6710 (1)	4125 (2)	1048 (1)	4.2
C(2)	6612 (1)	5515 (2)	1618 (1)	4.0
B(3)	7133 (1)	6216 (2)	1094 (1)	3.3
B(4)	6754 (1)	5360 (3)	397 (1)	4.2
B(5)	6055 (1)	4082 (3)	550 (1)	4.8
B(6)	5981 (1)	4147 (3)	1340 (1)	4.6
B(7)	6579 (1)	7797 (2)	1401 (1)	4.0
B(8)	6657 (1)	7741 (3)	610 (1)	4.1
B(9)	5998 (1)	6430 (3)	273 (1)	4.8
B(10)	5520 (1)	5676 (3)	859 (1)	4.6
B(11)	5880 (1)	6536 (3)	1553 (1)	4.6
B(12)	5891 (1)	7946 (3)	896 (1)	4.8
B(1')	9007 (1)	5420 (2)	2103 (1)	4.0
B(2')	9041 (1)	3594 (3)	1570 (1)	4.4
B(3')	8292 (1)	4428 (2)	1750 (1)	3.6
B(4')	8318 (1)	6871 (2)	1999 (1)	3.7
C(5')	9429 (1)	5694 (2)	1505 (1)	4.4
C(6')	9126 (1)	4919 (2)	944 (1)	4.5
B(7')	8449 (1)	4264 (2)	971 (1)	4.0
B(8')	7920 (1)	6216 (2)	1286 (1)	3.2
B(9')	8333 (1)	8400 (2)	1391 (1)	3.9
B(10')	9074 (1)	7736 (2)	1772 (1)	4.4

Table 2. Fractional coordinates ( $\times 10^3$ ) and isotropic temperature factors for H atoms with e.s.d.'s in parentheses

	x	y	z	$B_{iso}$ (Å <sup>2</sup> )
H(C1)	695 (1)	302 (3)	116 (1)	6.2 (4)
H(C2)	680 (1)	512 (2)	200 (1)	4.9 (3)
H(B4)	708 (1)	491 (3)	9 (1)	7.0 (4)
H(B5)	596 (1)	270 (3)	33 (1)	6.8 (4)
H(B6)	586 (1)	293 (3)	162 (1)	6.7 (4)
H(B7)	678 (1)	891 (2)	171 (1)	6.2 (4)
H(B8)	690 (1)	893 (2)	39 (1)	6.0 (4)
H(B9)	580 (1)	675 (2)	-18 (1)	5.7 (4)
H(B10)	500 (1)	540 (3)	79 (1)	6.7 (4)
H(B11)	566 (1)	671 (3)	201 (1)	6.5 (4)
H(B12)	561 (1)	930 (3)	85 (1)	6.4 (4)
H(B1')	927 (1)	526 (2)	257 (1)	4.7 (3)
H(B2')	931 (1)	227 (2)	168 (1)	5.4 (4)
H(B3')	796 (1)	338 (2)	197 (1)	5.1 (4)
H(B4')	808 (1)	731 (2)	238 (1)	5.1 (4)
H(C5')	987 (1)	570 (2)	156 (1)	5.8 (4)
H(C6')	941 (1)	456 (3)	60 (1)	6.6 (4)
H(B7')	821 (1)	332 (2)	64 (1)	5.3 (4)
H(B9')	810 (1)	978 (2)	133 (1)	5.0 (3)
H(B10')	940 (1)	872 (3)	201 (1)	6.4 (4)
H <sub>b</sub> (8',9')	817 (1)	738 (2)	93 (1)	5.2 (4)
H <sub>b</sub> (9',10')	893 (1)	842 (2)	126 (1)	3.0 (3)

Scattering factors were taken from *International Tables for X-ray Crystallography* (1974), except for H (Stewart, Davidson & Simpson, 1965).

**Discussion.** Fig. 1. shows a view of the iso-C<sub>4</sub>H<sub>22</sub>B<sub>18</sub> molecule. Bond lengths and selected bond angles are given in Table 3. The X-ray study confirms the suggestion that the structure consists of two different carbaborane frameworks, *i.e.* *nido*-5,6-C<sub>2</sub>H<sub>11</sub>B<sub>8</sub> and *closo*-1,2-C<sub>2</sub>H<sub>11</sub>B<sub>10</sub>. These two skeletons are bonded together by a B—B bond of 1.681 (2) Å between B(8') of the *nido* moiety and B(3) of the *closo*-1,2-dicarbaborane. The connecting bond retains the direction corresponding to that of the original terminal H atoms in both fragments. The mutual orientation of the two frameworks can be defined by the torsion angles C(1)—B(3)—B(8')—B(7') = 39.8, C(2)—B(3)—B(8')—B(7') = 105.6 and C(2)—B(3)—B(8')—B(3') = 38.1°.

The 5,6-dicarba-*nido*-decaborane framework, C<sub>2</sub>H<sub>11</sub>B<sub>8</sub>, resembles the decaborane(14) cage (Kasper, Lucht & Harker, 1950); however, it is substantially distorted due to the two C heteroatoms in the 5' and 6' positions. Between these C atoms a very short distance, 1.457 (2) Å, was found. The C—B distances in this part of the molecule range from 1.508 (2) to 1.751 (2) Å, the average B—B distance being 1.793 (47) Å. For the terminal H atoms, mean distances are B—H = 1.10 (3) and C—H = 0.98 (3) Å. There are also two bridging H atoms, between B(8') and B(9') and between B(9') and B(10'), with the B—H<sub>b</sub>—B angles both 88.4 (9)° and average B—H<sub>b</sub> distance 1.28 (3) Å.

The bond lengths in the 1,2-dicarbaborane icosahedron, C<sub>2</sub>H<sub>11</sub>B<sub>10</sub>, agree well with those in related icosahedral carbaboranes (Šubrtová *et al.*, 1980). The average B—B and B—C distances are 1.774 (9) and 1.708 (16) Å; B—H distances range from 1.04 (2) to

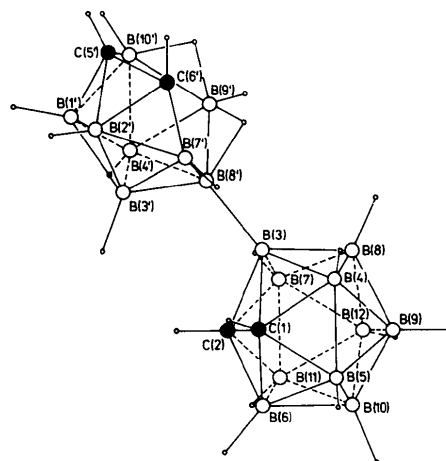


Fig. 1. Structure and numbering of the iso-C<sub>4</sub>H<sub>22</sub>B<sub>18</sub> molecule.

1.14 (2) Å and the C—H bond lengths are both 0.95 (2) Å. The shortest intermolecular H—H distance is 2.49 (2) Å.

Table 3. Bond distances (Å) and angles (°)

(a) Distances in the *nido*-C<sub>2</sub>H<sub>11</sub>B<sub>8</sub> moiety

B(1')—B(2')	1.752 (3)	B(3')—B(8')	1.774 (2)
B(1')—B(3')	1.789 (2)	B(4')—B(8')	1.788 (2)
B(1')—B(4')	1.782 (2)	B(4')—B(9')	1.727 (2)
B(1')—C(5')	1.657 (2)	B(4')—B(10')	1.814 (2)
B(1')—B(10')	1.805 (2)	C(5')—C(6')	1.457 (2)
B(2')—B(3')	1.758 (2)	C(5')—B(10')	1.751 (2)
B(2')—C(5')	1.709 (3)	C(6')—B(7')	1.508 (2)
B(2')—C(6')	1.691 (2)	B(7')—B(8')	1.937 (2)
B(2')—B(7')	1.813 (2)	B(8')—B(9')	1.782 (2)
B(3')—B(4')	1.815 (2)	B(8')—B(3)	1.681 (2)
B(3')—B(7')	1.777 (2)	B(9')—B(10')	1.783 (2)

(b) Selected angles in the *nido*-C<sub>2</sub>H<sub>11</sub>B<sub>8</sub> moiety involving C atoms

B(2')—B(1')—C(5')	60.1 (1)	B(1')—C(5')—B(10')	63.9 (1)
C(5')—B(1')—B(10')	60.6 (1)	B(2')—C(5')—C(6')	64.0 (1)
B(1')—B(2')—C(5')	57.2 (1)	B(2')—C(6')—C(5')	65.3 (1)
C(5')—B(2')—C(6')	50.8 (1)	B(2')—C(6')—B(7')	68.8 (1)
C(6')—B(2')—B(7')	50.9 (1)	B(2')—B(7')—C(6')	60.4 (1)
B(1')—C(5')—B(2')	62.7 (1)	B(1')—B(10')—C(5')	55.5 (1)

(c) Distances in the *closo*-C<sub>2</sub>H<sub>11</sub>B<sub>10</sub> moiety

C(1)—C(2)	1.622 (2)	B(5)—B(6)	1.764 (3)
C(1)—B(3)	1.728 (2)	B(5)—B(9)	1.772 (3)
C(1)—B(4)	1.690 (2)	B(5)—B(10)	1.769 (3)
C(1)—B(5)	1.701 (2)	B(6)—B(10)	1.758 (3)
C(1)—B(6)	1.712 (2)	B(6)—B(11)	1.773 (3)
C(2)—B(3)	1.728 (2)	B(7)—B(8)	1.765 (3)
C(2)—B(6)	1.721 (2)	B(7)—B(11)	1.778 (2)
C(2)—B(7)	1.685 (2)	B(7)—B(12)	1.767 (2)
C(2)—B(11)	1.701 (2)	B(8)—B(9)	1.787 (3)
B(3)—B(4)	1.785 (2)	B(8)—B(12)	1.786 (2)
B(3)—B(7)	1.788 (2)	B(9)—B(10)	1.780 (2)
B(3)—B(8)	1.776 (2)	B(9)—B(12)	1.772 (3)
B(4)—B(5)	1.783 (3)	B(10)—B(11)	1.766 (3)
B(4)—B(8)	1.766 (3)	B(10)—B(12)	1.787 (3)
B(4)—B(9)	1.768 (2)	B(11)—B(12)	1.762 (3)

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## 1,4-Bis(isopropylamino)anthraquinone

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**Abstract.** C<sub>20</sub>H<sub>22</sub>N<sub>2</sub>O<sub>2</sub>,  $M_r = 322.18$ , trigonal,  $R\bar{3}$ ,  $a = 15.030$  (5) Å,  $\alpha = 106.4$  (1)°,  $V = 2872.65$  Å<sup>3</sup>,  $D_m = 1.165$ ,  $D_c = 1.118$  Mg m<sup>-3</sup>,  $Z = 6$ ,  $\lambda$  (Mo) = 0.71069 Å,  $\mu$ (Mo  $K\alpha$ ) = 0.079 mm<sup>-1</sup>,  $F(000) = 1032$ ,

0567-7408/82/123149-03\$01.00

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$R = 0.056$  for 2307 observed reflexions. The structure comprises discrete molecules with two internal hydrogen bonds N—H...O per molecule but no intermolecular interactions other than van der Waals forces.

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