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## Structure of 8-Dimethylsulphido-7,9-dicarba-*nido*-undecaborane(11),\* C<sub>4</sub>H<sub>17</sub>B<sub>9</sub>S

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**Abstract.**  $M_r = 194.6$ ,  $P2_1/c$ ,  $a = 10.043$  (4),  $b = 8.936$  (4),  $c = 12.946$  (6) Å,  $\beta = 94.48$  (3)°,  $V = 1158.3$  (9) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.116$  Mg m<sup>-3</sup>,  $Cu K\alpha$ ,  $\lambda = 1.54178$  Å,  $\mu = 1.94$  mm<sup>-1</sup>,  $F(000) = 408$ , room temperature,  $R = 0.071$  for 1368 observed independent reflections. The S(CH<sub>3</sub>)<sub>2</sub> group, which has normal bond lengths and angles, is bonded to the B atom located between two C atoms in an open face of the C<sub>2</sub>B<sub>9</sub> *nido*-carbaborane cage. Centroids of the cages have twelve nearest neighbours and simulate a distorted cubic close packing.

**Introduction.** X-ray investigation of the title compound, which forms part of our continuing studies on structures of the thio derivatives of the carbaboranes, has been undertaken in order to clarify the position of the S(CH<sub>3</sub>)<sub>2</sub> group on the 7,9-C<sub>2</sub>B<sub>9</sub> *nido* skeleton. Crystals were prepared in the Institute of Inorganic Chemistry, Czechoslovak Academy of Sciences (Plešek, Janoušek & Heřmánek, 1978).

**Experimental.** Transparent colourless crystal 0.35 × 0.30 × 0.40 mm, Syntex  $P2_1$  diffractometer,  $Cu K\alpha$  radiation, graphite monochromator. Cell parameters and standard deviations by least squares from 23 high-order reflections. 1574 independent reflections measured by  $\omega/2\theta$  scans to  $2\theta = 115^\circ$  (for  $h 0 \rightarrow 10$ ,  $k 0 \rightarrow 9$ ,  $l -14 \rightarrow +14$ ). 1368 reflections with  $I > 1.96\sigma(I)$  regarded as observed [ $\sigma(I)$  from counting statistics]. Intensities of three standards measured every 25 reflections showed insignificant changes. Data scaled according to standard reflections and corrected for Lorentz and polarization effects, but not for absorption

or extinction. Structure solved by direct methods with *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974), all non-H atoms located on *E* map. C cage atoms distinguished from B atoms by their lower temperature factors and shorter bond distances. Refinement with local version of full-matrix least-squares program *ORFLS* (Busing, Martin & Levy, 1962), anisotropic temperature factors for non-H atoms. H atoms from difference Fourier synthesis, refined with individual isotropic temperature factors. Function minimized  $\sum w(|F_o| - |F_c|)^2$  with  $w^{-1} = \sigma^2(F_o) + (0.015F_o)^2$ . In final cycle  $R = 0.071$ ,  $R_w = 0.088$ ,  $av. \Delta/\sigma = 0.07$ ,  $max. = 0.47$  for H atom in methyl group. Max. and min. heights in final  $\Delta\rho$  map +0.36 and -0.60 e Å<sup>-3</sup>. Scattering factors from *International Tables for X-ray Crystallography* (1974) except for H (Stewart, Davidson & Simpson, 1965). Calculations performed on IBM 370/135 and Siemens 7536 computers.

**Discussion.** The positional parameters are in Table 1, † bond lengths and selected angles in Table 2. The molecular structure is shown in Fig. 1. The orientation of the S(CH<sub>3</sub>)<sub>2</sub> group [bonded to B(8) in an open face of the *nido*-carbaborane cage] is defined by the torsion angles given in Table 2. The bond distances B–S = 1.886 (4), average S–C = 1.790 (6) Å and B–S–C and C–S–C angles are in good agreement with those found for other S(CH<sub>3</sub>)<sub>2</sub> groups in related borane

† Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and a unit-cell packing diagram have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39584 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

\* 8-Dimethylsulphonio-7,9-dicarba-*nido*-undecaborate(1-).

compounds (Mizusawa, Rudnick & Eriks, 1980; Schramm & Ibers, 1977). The deviations of the five atoms C(7)–B(8)–C(9)–B(10)–B(11) from their mean plane range from 0.048 (5) to 0.083 (4) Å. The mean B–C and B–B distances are 1.65 (3) and 1.78 (2) Å, respectively. The longest B–B bond, B(10)–B(11) = 1.841 (6) Å, occurs between the atoms to which the bridging hydrogen is symmetrically bonded with B–H = 1.28 (3) Å and B–H–B = 91 (2)°. A view of the molecular packing is shown in Fig. 2. Each C<sub>2</sub>B<sub>9</sub> cage has twelve nearest neighbours. The distances of the centroids of these cages (calculated from the vertices) range from 6.53 (1) to 8.94 (1) Å. Centroids form slightly corrugated plane layers parallel to (10 $\bar{2}$ ) and simulate a distorted cubic close packing as viewed along [10 $\bar{1}$ ]. Here the (10 $\bar{2}$ ) planes correspond to the cubic (111). In previous work (Novák, Šubrtová, Líněk & Hašek, 1983) we have described another case of simulation of close packing by carbaborane cages.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{Å}^2)$
S	3344.8 (8)	4496.0 (9)	9328.5 (7)	4.59 (3)
C(1)	1954 (6)	3723 (6)	9924 (5)	6.3 (2)
C(2)	3332 (7)	3442 (5)	8148 (5)	6.5 (2)
B(1)	3123 (4)	9058 (5)	7821 (3)	4.7 (1)
B(2)	1572 (4)	8258 (5)	7447 (3)	4.6 (1)
B(3)	2973 (4)	7087 (5)	7644 (3)	4.3 (1)
B(4)	3980 (4)	7790 (4)	8707 (3)	3.9 (1)
B(5)	3200 (4)	9395 (4)	9157 (3)	4.7 (1)
B(6)	1661 (4)	9658 (4)	8414 (3)	4.4 (1)
C(7)	1479 (4)	6668 (4)	8093 (3)	4.3 (1)
B(8)	2756 (4)	6423 (4)	8912 (2)	3.3 (1)
C(9)	3021 (4)	7739 (4)	9720 (2)	4.2 (1)
B(10)	1678 (5)	8791 (5)	9659 (3)	5.0 (1)
B(11)	623 (4)	8054 (5)	8547 (3)	4.7 (1)

Table 2. Bond distances (Å) and selected angles (°) and torsion angles (°)

S–B(8)	1.886 (4)	B(3)–B(8)	1.775 (5)
S–C(1)	1.786 (6)	B(4)–B(5)	1.755 (5)
S–C(2)	1.794 (6)	B(4)–B(8)	1.768 (5)
B(1)–B(2)	1.748 (6)	B(4)–C(9)	1.687 (5)
B(1)–B(3)	1.781 (6)	B(5)–B(6)	1.771 (5)
B(1)–B(4)	1.785 (6)	B(5)–C(9)	1.666 (5)
B(1)–B(5)	1.751 (6)	B(5)–B(10)	1.790 (6)
B(1)–B(6)	1.791 (6)	B(6)–B(10)	1.787 (6)
B(2)–B(3)	1.756 (6)	B(6)–B(11)	1.789 (6)
B(2)–B(6)	1.767 (6)	C(7)–B(8)	1.614 (5)
B(2)–C(7)	1.655 (6)	C(7)–B(11)	1.642 (6)
B(2)–B(11)	1.784 (6)	B(8)–C(9)	1.583 (5)
B(3)–B(4)	1.759 (5)	C(9)–B(10)	1.641 (6)
B(3)–C(7)	1.693 (6)	B(10)–B(11)	1.841 (6)
C(7)–B(8)–S	121.8 (2)	B(8)–S–C(1)	103.8 (2)
C(9)–B(8)–S	117.2 (2)	B(8)–S–C(2)	104.8 (2)
		C(1)–S–C(2)	102.2 (3)
C(7)–B(8)–S–C(1)	–61.2 (3)		
C(7)–B(8)–S–C(2)	45.6 (4)		
C(9)–B(8)–S–C(1)	89.3 (3)		
C(9)–B(8)–S–C(2)	–163.9 (3)		

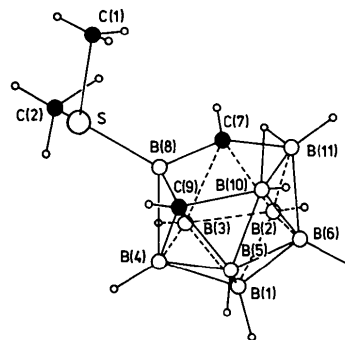


Fig. 1. Structure and numbering of  $(\text{CH}_3)_2\text{SC}_2\text{B}_9\text{H}_{11}$

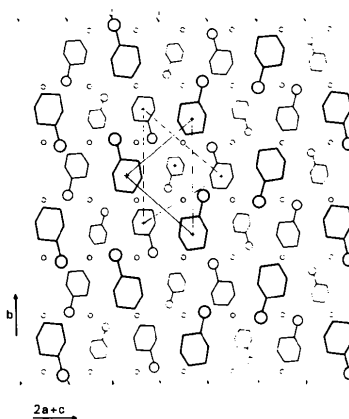


Fig. 2. View along  $[10\bar{1}]$  of the layers parallel to  $(10\bar{2})$ . The three sizes of the graphic symbols represent the molecules corresponding to the first, second and third layers of the packing. The relative position of the layers is emphasized by the triangles. The symmetry elements are shown.

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