Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Sn—S(1)	2.526 (1)	N(2)—C(12)	1.490 (7)
Sn—S(2)	3.021 (2)	C(2)—C(3)	1.536 (7)
Sn—S(3)	2.528 (1)	C(3)—C(4)	1.501 (7)
Sn—S(4)	2.937 (1)	C(5)—C(6)	1.510 (7)
Sn—C(15)	2.132 (5)	C(6)—C(7)	1.478 (8)
Sn—C(19)	2.163 (6)	C(9)—C(10)	1.514 (8)
S(1)—C(1)	1.743 (4)	C(10)—C(11)	1.518 (9)
S(2)—C(1)	1.694 (4)	C(12)—C(13)	1.477 (11)
S(3)—C(8)	1.745 (4)	C(13)—C(14)	1.561 (11)
S(4)—C(8)	1.682 (4)	C(15)—C(16)	1.542 (6)
N(1)—C(1)	1.334 (5)	C(16)—C(17)	1.507 (8)
N(1)—C(2)	1.457 (5)	C(17)—C(18)	1.489 (9)
N(1)—C(5)	1.478 (5)	C(19)—C(20)	1.329 (14)
N(2)—C(8)	1.337 (6)	C(20)—C(21)	1.640 (18)
N(2)—C(9)	1.473 (6)	C(21)—C(22)	1.419 (16)
S(1)—Sn—S(2)	63.9 (1)	S(1)—C(1)—N(1)	117.6 (3)
S(1)—Sn—S(3)	82.4 (1)	S(2)—C(1)—N(1)	123.1 (3)
S(1)—Sn—S(4)	147.4 (1)	S(3)—C(8)—N(2)	117.4 (3)
S(2)—Sn—S(3)	145.9 (1)	S(4)—C(8)—N(2)	123.0 (3)
S(2)—Sn—S(4)	148.6 (1)	C(1)—N(1)—C(2)	122.4 (3)
S(3)—Sn—S(4)	65.1 (1)	C(1)—N(1)—C(5)	121.1 (3)
C(15)—Sn—C(19)	132.6 (2)	C(8)—N(2)—C(9)	123.6 (4)
C(15)—Sn—S(1)	107.5 (1)	C(8)—N(2)—C(12)	121.1 (4)
C(15)—Sn—S(2)	82.1 (1)	N(1)—C(2)—C(3)	112.4 (4)
C(15)—Sn—S(3)	104.4 (1)	N(1)—C(5)—C(6)	112.3 (4)
C(15)—Sn—S(4)	83.6 (1)	N(2)—C(9)—C(10)	110.9 (4)
C(19)—Sn—S(1)	106.3 (2)	N(2)—C(12)—C(13)	110.0 (5)
C(19)—Sn—S(2)	83.9 (2)	C(2)—N(1)—C(5)	116.4 (3)
C(19)—Sn—S(3)	112.3 (2)	C(9)—N(2)—C(12)	115.0 (4)
C(19)—Sn—S(4)	85.5 (2)	C(2)—C(3)—C(4)	112.2 (4)
Sn—S(1)—C(1)	95.9 (1)	C(5)—C(6)—C(7)	113.0 (5)
Sn—S(2)—C(1)	80.6 (2)	C(9)—C(10)—C(11)	110.9 (5)
Sn—S(3)—C(8)	93.7 (2)	C(12)—C(13)—C(14)	107.8 (6)
Sn—S(4)—C(8)	81.6 (2)	C(15)—C(16)—C(17)	112.3 (4)
Sn—C(15)—C(16)	112.8 (3)	C(16)—C(17)—C(18)	113.1 (5)
Sn—C(19)—C(20)	120.5 (7)	C(19)—C(20)—C(21)	124.3 (11)
S(1)—C(1)—S(2)	119.3 (2)	C(20)—C(21)—C(22)	114.8 (10)
S(3)—C(8)—S(4)	119.6 (3)		

square-planar geometry (*cis* S—Sn—S angles range from 63.9 to 148.6°). The small (but significant) deviations from planarity of the Sn—S_2CN ligand fragments are indicative of a small twist (< 2°) about the C—S bonds.

The crystal structure consists of relatively isolated molecules; all intermolecular distances are greater than 3.6 Å. The abnormal bond lengths and angles in one butyl ligand [C(20)—C(21)—C(22)] may be caused by the high thermal motion of corresponding atoms. The other C—C distances and angles are in keeping with those expected for single bonds.

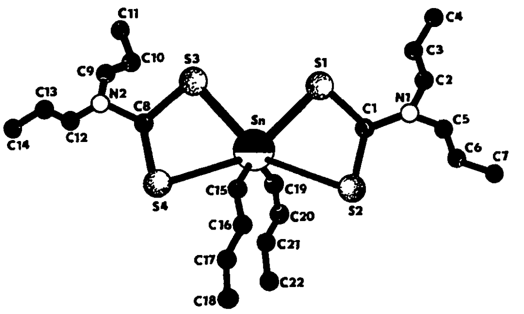

Fig. 1. Molecular structure of $[(n\text{-Bu})_2\text{Sn}(S_2\text{CNPr}_2)_2]$ showing the atomic numbering scheme.

The bond angles in the dtc ligands are not unusual and are in keeping with near sp^2 hybridization of the N atoms, and C(1) and C(8). There is the usual contraction from 120° of the angles opposite the C(1)—N(1) and C(8)—N(2) bonds indicative of the high multiple bond character in these bonds. The C(1)—N(1) and C(8)—N(2) distances of 1.334 (5) and 1.337 (6) Å respectively are intermediate between the sum of the single-bond radii (Pauling, 1960) and the sum of the double-bond radii, 1.29 Å.

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5-(η-*p*-Cymene)-6-ethoxy-5-ruthena-*nido*-decaborane(12)

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Abstract. (1) $C_{12}H_{31}B_9ORu$, $M_r = 389.3$, orthorhombic, $P2_12_12_1$, $a = 11.310$ (3), $b = 18.146$ (11), $c = 18.926$ (4) Å, $V = 3884$ (3) Å^3, two independent molecules per asymmetric unit, $Z = 8$, $D_x = 1.331$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.786$ mm^{-1}, $F(000) = 1600$, $T = 291$ (1) K, $R =$

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0.0254 for 3578 independent observed reflections. The title compound is formed by the reaction of $[6\text{-}(\eta\text{-}p\text{-cymene})\text{-}6\text{-}RuB_9H_{13}]$ with ethanolic NaOH. A crystallographic study shows the presence of an *endo*-hydrogen atom at the B(7) position and ^{11}B NMR spectroscopy indicates this structure is retained in solution. A series of EHMO (extended Hückel molecular orbital) calculations suggests that the *endo*-hydrogen is a result of both the terminal ligand substitution at B(6) and the metallatropic shift of the Ru atom.

Introduction. Many metal-containing analogues of *nido*-$B_{10}H_{14}$ are known (Kennedy, 1986). The reaction of one such species, $[6\text{-}(\eta\text{-}p\text{-cymene})RuB_9H_{13}]$, made by a similar route to that which affords $[6\text{-}(\eta\text{-}C_6Me_6)RuB_9H_{13}]$ (Bown, Greenwood & Kennedy, 1986), was studied in an attempt to synthesize a metallaborane analogue of *arachno*-$[B_9H_{14}]^-$. A major product of this reaction was the title compound, the crystallographic analysis of which is presented here.

Experimental. $[5\text{-}(\eta\text{-}C_{10}H_{14})\text{-}6\text{-OEt-}5\text{-}RuB_9H_{12}]$ (1), isolated in 34% yield *via* column chromatography [fluorasil (100–200 mesh), 8 × 1.5 cm, CH_2Cl_2 eluent] of the product from a room-temperature reaction of $[6\text{-}(\eta\text{-}C_{10}H_{14})RuB_9H_{13}]$ with NaOH in ethanol. The product is an ochre solid from which golden-yellow crystals were grown by slow diffusion of *n*-hexane into a concentrated CH_2Cl_2 solution at 243 K. NMR ($CDCl_3$, 291 K) $^{11}B(^1H)$ δ 44.76, 15.23, 10.13, −0.37, −4.45 (2*B*, coincidental), −15.45, −17.45 and −42.93. Crystal (0.4 × 0.15 × 0.1 mm) mounted in a sealed Lindemann capillary on an Enraf–Nonius CAD-4 diffractometer (Mo $K\alpha$ radiation, graphite monochromator); cell parameters and orientation matrix from least-squares refinement ($14 < \theta < 15°$) of 25 centred reflections; data collection by ω–2θ scans in 96 steps with ω-scan width $(0.8 + 0.34\tan\theta)°$; data (*h*: 0 to 13, *k*: 0 to 21, *l*: 0 to 22) measured for $1 \leq \theta \leq 25°$ over 84 X-ray hours; no noticeable crystal movement or decay; corrections made for Lorentz and polarization effects (Gould & Smith, 1986) and, following isotropic convergence, absolute configuration was established by parallel refinement of both enantiomers, the model used having $R = 0.0509$ at this stage, the other enantiomeric form, $R = 0.0518$; empirical absorption correction (Walker & Stuart, 1983) applied (correction factors 0.954–1.108); 3854 data measured of which 3578 $[F \geq 2.0\sigma(F)]$ were retained; structure solution *via* Patterson synthesis (Ru) and iterative full-matrix least-squares refinement (on F)/ΔF syntheses (all other atoms from *SHELX*76; Sheldrick, 1976); all heavy atoms refined allowing full anisotropic thermal motion; weights assigned according to $w^{-1} = [\sigma^2(F) + 0.000660F^2]$; model refined in two blocks, corresponding to the two independent molecules (*A* and *B*) present, total number of variables 517; data: variables > 6.8; cage and phenyl H atoms located and positionally refined with group isotropic thermal parameters $[U_{\text{cage-H}(A)} = 0.055\,(4)$, $U_{\text{cage-H}(B)} = 0.045\,(4)$, $U_{\text{phenyl-H}(A)} = 0.038\,(6)$ and $U_{\text{phenyl-H}(B)} = 0.044\,(6)\,\text{Å}^2]$; all other H atoms set in idealized positions with CH_3 moieties being treated as rigid groups and assigned a single group isotropic thermal parameter $[U_{\text{alkyl-H}(A)} = 0.095\,(5)$ and $U_{\text{alkyl-H}(B)} = 0.116\,(6)\,\text{Å}^2]$; max. shift/e.s.d. in final cycle < 0.02; $R = 0.0254$, $wR = 0.0321$, $S = 0.940$; max. and min. values in final ΔF synthesis 0.18 and $-0.20\,\text{e}\,\text{Å}^{-3}$ respectively; scattering factors for C, H, B and O inlaid in *SHELX*76 (Sheldrick, 1976); those for Ru taken from *International Tables for X-ray Crystallography* (1974, Vol. IV); figure drawn using *EASYORTEP* (Mallinson & Muir, 1985) and geometrical calculations used *CALC* (Gould & Taylor, 1986).

Discussion. Table 1* lists the coordinates of heavy atoms with equivalent isotropic thermal parameters for molecules *A* and *B*. Table 2 compares selected interatomic distances and interbond angles in each molecule. A perspective view of each molecule (Fig. 1) clearly shows that the crystal selected for analysis contains a racemic mixture of two enantiomeric forms of (1). The common configuration adopted by the *p*-cymene moiety in both molecules, with the C(7) methyl lying *cis* to the ethoxy group, can also be seen. Whilst there is some evidence for a weak hydrogen-bonding interaction in molecule *A* [HC(72)—O = 2.539 (6) Å] this is not apparent in molecule *B* (no H—O distances < 3 Å). The observed preferred conformation may instead be a result of the different *trans* influences exerted on the *p*-cymene moiety by the borane cage.

The structure of the substituted ruthenaborane is related to the of $B_{10}H_{14}$ *via* the isolobal replacement, at the 5 position, of a {BH} fragment with a {Ru(*p*-cymene)} moiety and substitution of a terminal H atom with an ethoxy group at B(6). During the course of the reaction a cluster rearrangement occurs resulting in the {Ru(*p*-cymene)} moiety moving from the 6 to the 5 position. Such metallatropic shifts are known for other metallaborane systems, especially in the presence of nucleophiles. For example, treatment of [6-(η-

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* Lists of structure factors, anisotropic thermal parameters, full interatomic distances and interbond angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54659 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0284]

Table 1. *Fractional coordinates of non-*H *atoms and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Molecule *A*				
Ru(5)	−0.13431 (3)	−0.06967 (2)	−0.19864 (1)	0.0286 (1)
B(1)	−0.0634 (4)	0.0413 (3)	−0.1783 (3)	0.0383 (25)
B(6)	−0.1653 (4)	−0.0343 (3)	−0.30567 (25)	0.0387 (25)
B(10)	−0.2056 (5)	0.0296 (3)	−0.1399 (3)	0.040 (3)
B(4)	−0.1605 (5)	0.11914 (25)	−0.1693 (3)	0.042 (3)
B(8)	−0.2245 (5)	0.1314 (3)	−0.2548 (3)	0.044 (3)
B(7)	−0.1474 (5)	0.0646 (3)	−0.3200 (3)	0.0426 (25)
B(9)	−0.3033 (5)	0.0956 (3)	−0.1800 (3)	0.044 (3)
B(2)	−0.0434 (4)	0.01251 (25)	−0.2670 (3)	0.0359 (25)
B(3)	−0.0773 (5)	0.1061 (3)	−0.2470 (3)	0.043 (3)
C(1)	−0.0176 (4)	−0.16770 (22)	−0.22459 (25)	0.0419 (24)
C(2)	−0.1399 (4)	−0.19066 (21)	−0.21996 (23)	0.0405 (21)
C(5)	−0.0408 (4)	−0.11516 (23)	−0.10498 (25)	0.0435 (25)
C(3)	−0.2039 (4)	−0.17881 (21)	−0.15931 (24)	0.0367 (23)
C(4)	−0.1591 (4)	−0.14188 (21)	−0.09947 (21)	0.0379 (22)
C(6)	0.0286 (4)	−0.12769 (23)	−0.1654 (3)	0.0437 (25)
C(7)	0.0554 (4)	−0.1809 (3)	−0.2897 (3)	0.056 (3)
C(8)	−0.2270 (4)	−0.1321 (3)	−0.03220 (22)	0.0476 (25)
C(9)	−0.3576 (5)	−0.1320 (3)	−0.0418 (3)	0.067 (3)
C(10)	−0.1895 (5)	−0.1937 (3)	0.0189 (3)	0.068 (3)
O	−0.1740 (3)	−0.08335 (16)	−0.36133 (14)	0.0446 (16)
CO(1)	−0.1451 (6)	−0.0619 (3)	−0.43222 (22)	0.065 (3)
CO(2)	−0.1420 (5)	−0.1283 (3)	−0.4781 (3)	0.068 (3)
Molecule *B*				
Ru(5)	−0.12901 (3)	−0.41958 (1)	−0.61044 (1)	0.0300 (1)
B(6)	−0.1574 (4)	−0.4543 (3)	−0.71807 (24)	0.0376 (24)
B(10)	−0.1941 (5)	−0.5216 (3)	−0.5539 (3)	0.043 (3)
B(1)	−0.0514 (4)	−0.5296 (3)	−0.5915 (3)	0.041 (3)
B(2)	−0.0341 (5)	−0.49893 (25)	−0.6805 (3)	0.040 (3)
B(7)	−0.1346 (5)	−0.5510 (3)	−0.7348 (3)	0.044 (3)
B(3)	−0.0618 (5)	−0.5929 (3)	−0.6628 (3)	0.046 (3)
B(4)	−0.1434 (6)	−0.6101 (3)	−0.5847 (3)	0.049 (3)
B(9)	−0.2901 (5)	−0.5893 (3)	−0.5950 (3)	0.052 (3)
B(8)	−0.2095 (5)	−0.6209 (3)	−0.6705 (3)	0.048 (3)
C(1)	−0.0231 (4)	−0.31691 (24)	−0.63618 (25)	0.0457 (25)
C(2)	0.0293 (4)	−0.3570 (3)	−0.5791 (3)	0.049 (3)
C(3)	−0.0338 (4)	−0.37350 (25)	−0.51754 (25)	0.0433 (24)
C(4)	0.1537 (4)	0.35124 (22)	−0.50923 (22)	0.0398 (22)
C(5)	−0.2071 (4)	−0.31494 (24)	−0.56602 (23)	0.0396 (23)
C(6)	−0.1425 (4)	−0.29784 (22)	−0.62867 (23)	0.0428 (23)
C(7)	0.0468 (5)	−0.2992 (3)	−0.7008 (3)	0.066 (3)
C(8)	−0.2168 (4)	−0.3682 (3)	−0.44074 (23)	0.048 (3)
C(9)	−0.1788 (5)	−0.3148 (3)	−0.38274 (24)	0.063 (3)
C(10)	−0.3516 (5)	−0.3695 (4)	−0.4482 (3)	0.074 (4)
O	−0.1723 (3)	−0.40398 (17)	−0.77145 (15)	0.0455 (16)
CO(1)	−0.1407 (5)	−0.4218 (3)	−0.84312 (22)	0.060 (3)
CO(2)	−0.1510 (5)	−0.3534 (3)	−0.8869 (3)	0.071 (4)

Table 2. *Comparison of selected interatomic distances* (Å) *and angles* (°)

	A	*B*
Ru(5)—B(1)	2.202 (5)	2.210 (5)
Ru(5)—B(2)	2.226 (5)	2.232 (5)
Ru(5)—B(6)	2.153 (5)	2.156 (5)
Ru(5)—B(10)	2.264 (5)	2.262 (5)
Ru(5)—H(56)	1.59 (5)	1.65 (4)
Ru(5)—C(1)	2.269 (4)	2.268 (5)
Ru(5)—C(2)	2.232 (4)	2.202 (5)
Ru(5)—C(3)	2.257 (4)	2.225 (5)
Ru(5)—C(4)	2.306 (4)	2.299 (4)
Ru(5)—C(5)	2.223 (5)	2.257 (5)
Ru(5)—C(6)	2.213 (5)	2.241 (4)
B(1)—B(2)	1.773 (7)	1.784 (7)
B(1)—B(3)	1.760 (7)	1.775 (8)
B(1)—B(4)	1.797 (7)	1.798 (8)
B(1)—B(10)	1.777 (7)	1.771 (8)
B(1)—HB(1)	1.11 (5)	1.05 (4)
B(2)—B(3)	1.781 (7)	1.766 (8)
B(2)—B(6)	1.777 (7)	1.762 (7)
B(2)—B(7)	1.812 (7)	1.800 (7)
B(2)—HB(2)	1.15 (5)	1.20 (4)
B(3)—B(4)	1.762 (7)	1.769 (8)
B(3)—B(7)	1.762 (8)	1.765 (8)
B(3)—B(8)	1.733 (8)	1.752 (8)
B(3)—HB(3)	1.16 (5)	0.96 (5)
B(4)—B(8)	1.785 (8)	1.798 (8)
B(4)—B(9)	1.683 (8)	1.712 (8)
B(4)—B(10)	1.792 (7)	1.801 (8)
B(4)—HB(4)	1.15 (4)	1.08 (4)
B(6)—B(7)	1.827 (7)	1.802 (7)
B(6)—H(56)	1.44 (5)	1.40 (4)
B(7)—B(8)	1.938 (8)	1.951 (8)
B(7)—HB(7)	1.25 (4)	1.05 (4)
B(7)—H(en)	0.94 (5)	1.23 (4)
B(8)—B(9)	1.794 (8)	1.789 (8)
B(8)—HB(8)	1.06 (5)	1.02 (4)
B(8)—H(89)	1.25 (5)	1.24 (4)
B(9)—B(10)	1.797 (8)	1.814 (8)
B(9)—HB(9)	0.90 (5)	1.12 (4)
B(9)—H(89)	1.27 (5)	1.14 (4)
B(9)—H(910)	1.28 (5)	1.24 (5)
B(10)—HB(10)	1.07 (5)	1.17 (4)
B(10)—H(910)	1.31 (5)	1.20 (5)
B(6)—O	1.382 (6)	1.372 (6)
O—CO(1)	1.435 (6)	1.440 (6)
CO(1)—CO(2)	1.486 (8)	1.497 (8)
B(1)—Ru(5)—B(2)	47.2 (2)	47.3 (2)
B(1)—Ru(5)—B(10)	46.9 (2)	46.6 (2)
B(2)—Ru(5)—B(6)	47.9 (2)	47.3 (2)
Ru(5)—B(1)—B(2)	67.1 (2)	67.0 (2)
Ru(5)—B(1)—B(10)	68.4 (2)	68.2 (3)
B(2)—B(1)—B(3)	60.5 (3)	59.5 (3)
B(3)—B(1)—B(4)	59.4 (3)	59.3 (3)
B(4)—B(1)—B(10)	60.2 (3)	60.6 (3)
Ru(5)—B(2)—B(1)	65.7 (2)	65.7 (2)
Ru(5)—B(2)—B(6)	63.9 (2)	64.1 (2)
B(1)—B(2)—B(3)	59.4 (3)	60.0 (3)
B(3)—B(2)—B(7)	58.7 (3)	59.3 (3)
B(6)—B(2)—B(7)	61.2 (3)	60.8 (3)
B(1)—B(3)—B(2)	60.1 (3)	60.5 (3)
B(1)—B(3)—B(4)	61.4 (3)	61.0 (3)
B(2)—B(3)—B(7)	61.5 (3)	61.3 (3)
B(4)—B(3)—B(8)	61.4 (3)	61.4 (3)
B(7)—B(3)—B(8)	67.3 (3)	67.4 (3)
B(1)—B(4)—B(3)	59.3 (3)	59.7 (3)
B(1)—B(4)—B(10)	59.4 (3)	58.9 (3)
B(3)—B(4)—B(8)	58.5 (3)	58.8 (3)
B(8)—B(4)—B(9)	62.2 (3)	61.2 (3)
B(9)—B(4)—B(10)	62.2 (3)	62.1 (3)
Ru(5)—B(6)—B(2)	68.2 (2)	68.6 (2)
B(2)—B(6)—B(7)	60.4 (3)	60.7 (3)
B(2)—B(6)—O	132.6 (4)	134.5 (4)
B(2)—B(7)—B(3)	59.8 (3)	59.4 (3)
B(2)—B(7)—B(6)	58.5 (3)	58.6 (3)
B(3)—B(7)—B(8)	55.6 (3)	56.0 (3)
B(3)—B(8)—B(4)	60.1 (3)	59.7 (3)
B(3)—B(8)—B(7)	57.0 (3)	56.6 (3)
B(4)—B(8)—B(9)	56.1 (3)	57.0 (3)
B(4)—B(9)—B(8)	61.7 (3)	61.8 (3)
B(4)—B(9)—B(10)	61.9 (3)	61.4 (3)
Ru(5)—B(10)—B(1)	64.7 (2)	65.2 (2)
B(1)—B(10)—B(4)	60.5 (3)	60.4 (3)
B(4)—B(10)—B(9)	55.9 (3)	56.5 (3)
B(6)—O—CO(1)	121.4 (4)	120.9 (4)
O—CO(1)—CO(2)	109.4 (4)	108.4 (4)

$C_6Me_6)RuB_9H_{13}]$ with phosphines (PMe_2Ph or PPh_3) gives metalladecaboranes with the {Ru(arene)} moiety at the 5 position and terminal hydrogen substitution, by phosphine, at the 7 position (Bown, Fontaine, Greenwood & Kennedy, 1987). A species closely related to the title compound, [5-(η-C_6Me_6)-6-OMe-5-RuB_9H_{12}] (2) (Bown, Fontaine, Greenwood, Kennedy & Thornton-Pett, 1988), is formed in the degradation-rearrangement reaction between $[B_{10}H_{10}]^{2-}$ and $[(p\text{-cymene})RuCl_2]_2$ in methanol. Comparison of the structures of (1) and (2) show them to be analogous except that, in the former, the B(6)—B(7) bond is not bridged, but rather B(7) carries an *endo*-H atom. Distances around this *endo*-H atom [H(en)—B(6) = 1.50 (6), H(en)—B(7) = 1.09 (7) Å]* and the B(6)—B(7) hydrogen bridge in (2) [H(67)—B(6) = 1.53 (3), H(67)—B(7) =

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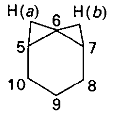
* Average distances for molecules *A* and *B* taken.

1.13 (3) Å] show them to be similarly positioned relative to their neighbouring heavy atoms. We believe that the B(6)—H(en) distance of 1.50 (6) Å in (1) is indeed too long to represent a true B—H bridging bond and that H(67) in (2) is also better described as an *endo*-H atom. Independent evidence for this view is found in the coupled ^{11}B NMR spectrum of (1) in which the resonance at $\delta = -15.45$, in addition to the doublet coupling due to the *exo*-H atom, exhibits an extra splitting of 43 Hz, characteristic of a $\{BH_2\}$ unit. Despite this unusual feature, the overall geometry and electron count of (1) adhere to that of a true *nido*-5-metalladecaborane.

Table 3. *ROM data for substituted boranes and metallaboranes*



Model	Atom pair: 6—H(*a*)	5—H(*a*)	6—H(*b*)	7—H(*b*)
(3) [5 = 6 = {BH}]	0.4250	0.4327		
(4) [5 = {BOH}, 6 = {BH}]	0.4386	0.4197		
(5) [5 = {BH}, 6 = {BOH}]	0.4120	0.4457		
(6) [5 = {BH}, 6 = {Co(C_5Me_5)}]	0.2586	0.3992		
(7) [5 = {(*p*-cymene)Ru}, 6 = {BH}]	0.3601	0.2518	0.4091	0.4253

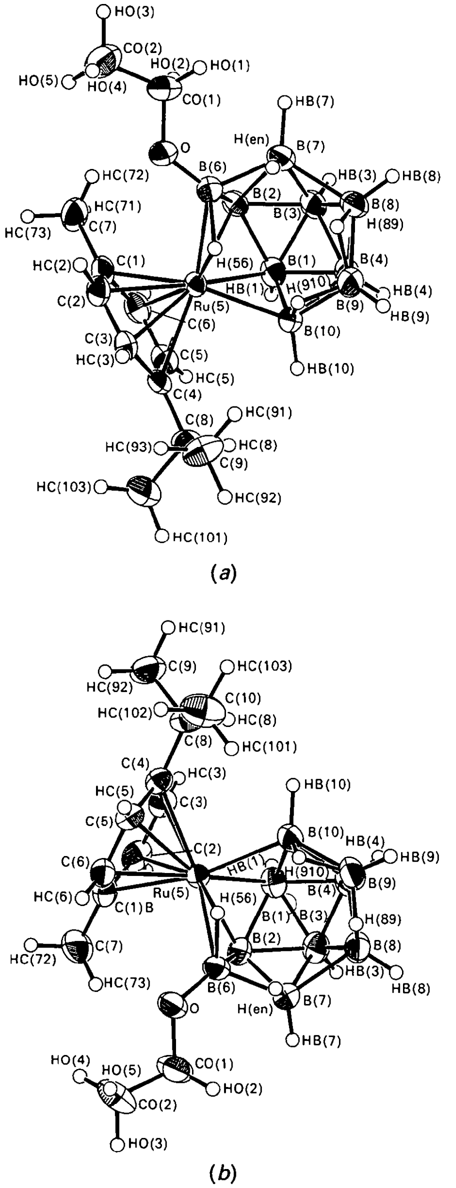


Fig. 1. Perspective views of (*a*) molecule *A* and (*b*) molecule *B*. Thermal ellipsoids are at 30% probability, except for H atoms which have an artificial radius of 0.1 Å.

The presence of the *endo*-H atom can be simply rationalized in terms of the influence of the (relatively) electronegative ethoxy group at B(6) which removes electron density from that site, reducing its attraction for an H^+ bridge. To analyse this interpretation more rigorously, a series of EHMO (extended Hückel molecular orbital) calculations using the *ICON*8 program (Howell, Rossi, Wallace, Haraki & Hoffmann, 1977) was undertaken on a number of substituted boranes and metallaboranes.

The models employed for this study were constructed such that the bridging H atoms near the site of substitution were placed symmetrically over the 5–6 and 6–7 connectivities. Inspection of the reduced overlap matrix (ROM) then gave an estimate of the strength of bonding between the hydrogen bridge and the neighbouring atoms, giving an indication towards which vertex any asymmetry should lie and, over a series of calculations, to what extent that asymmetry will present itself.

Three such symmetrized borane models were set up using average B(6)—B(5)—H_{bridge} and B(5)—B(6)—H_{bridge} angles and B(5,6)—H_{bridge} distances derived from the structure of $B_{10}H_{14}$ (Brill, Dietrich & Dierks, 1971). These were $B_{10}H_{14}$ (3), $B_{10}H_{13}$-5-OH (4) and $B_{10}H_{13}$-6-OH (5). The structures of two metalladecaboranes were similarly adapted: [6-(C_5Me_6)CoB_9H_{13}] (6) (Venable, Sinn & Grimes, 1982) and 5-(*p*-cymene)RuB_9H_{13} (7), derived from (1) by replacing the OEt group with an H atom. The results of the calculations on these species are shown in Table 3.

Starting with $B_{10}H_{14}$, the ROM reveals a slight asymmetry towards the more highly connected, and hence more positively charged, B atom, B(5). This small difference appears to be significant when the asymmetry observed in the structure of $B_{10}H_{14}$ is taken into account. Substitution with an electronegative group at the 6-position enhances this effect (5), whilst the influence of an OH substituent at B(5) is enough to reverse the asymmetry (4). Considering the metallaboranes, the presence of the $\{(C_5Me_5)Co\}$ moiety (6) favours bonding between the bridging H atom and B(5). However, as structural and solution NMR studies (Venable & Grimes, 1982) on this species show, this effect is not strong enough to cause any deviation in the pattern of bridging H atoms usually seen in $B_{10}H_{14}$ and its metallaborane analogues. Finally, in (7) when the metal is in the 5 position, a similar preference of the 5–6 bridging H atom towards the adjacent B atom is shown. However, a secondary effect is an increase [relative to (3)] in the asymmetry of the B(6)—B(7) bridging proton. It therefore appears that the observed *endo*-H atom at B(7) in the structure of (1) is a reflection of the directing influences of both the OEt substituent *and* the position of the metal.

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Structure of Di(*n*-butyl)bis(*N*,*N*-diethyldithiocarbamato)tin(IV) at 153 K

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Abstract. $[Sn(C_4H_9)_2(C_5H_{10}NS_2)_2]$, $M_r = 529.5$, monoclinic, Pc, $a = 12.434\,(2)$, $b = 11.913\,(3)$, $c = 18.822\,(4)$ Å, $\beta = 115.31\,(1)°$, $V = 2520.4\,(9)$ Å^3, $Z = 4$, $D_m = 1.39$, $D_x = 1.40$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.34$ mm^{-1}, $F(000) = 1096$, low temperature (153 K), final $R = 0.021$ for 4225 unique observed reflections. The structures of the two crystallographically independent molecules show only minor differences. The *N*,*N*-diethyldithiocarbamate (dtc) ligands in both molecules are chelated to tin in an unsymmetrical fashion, with one longer (2.90–3.05 Å) and one shorter (2.50–2.55 Å) Sn—S bond. The Sn atom has a distorted octahedral environment.

Introduction. Interest in dtc complexes of diorganotin species arises in part because of their varied structures (Morris & Schlemper, 1979*a*,*b*; Lindley & Carr, 1974; Lockhart, Manders & Schlemper, 1985; Lockhart, Manders, Schlemper & Zuckerman, 1986) and because of their biological activity (Bruckner, Hartel & Ger, 1961; Collins & Wiese, 1955). Crystallographic studies of these complexes have revealed a variety of coordination geometries around the Sn atom, ranging from tetrahedral to distorted octahedral. The coordination geometry depends on the bonding mode of the dtc moiety. In a continuing effort to understand the coordination chemistry of such complexes we have now determined the crystal structure of di(*n*-butyl)bis(*N*,*N*-diethyldithiocarbamato)tin(IV).

Experimental. The title compound was prepared by dissolving equimolar quantities of sodium *N*,*N*-diethyldithiocarbamate and di-*n*-butyltin dichloride

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