Table 2. *Bond distances (Å) and angles (°)*

	CSC		BCSC
	Molecule A	Molecule B	
O(1)—C(7)	1.231 (3)	1.237 (6)	1.237 (6)
N(3)—C(7)	1.320 (3)	1.331 (3)	1.330 (7)
N(2)—C(7)	1.367 (3)	1.363 (3)	1.363 (6)
N(2)—N(1)	1.382 (3)	1.382 (3)	1.393 (5)
N(1)—C(1)	1.278 (3)	1.277 (3)	1.278 (6)
C(1)—C(2)	1.491 (3)	1.493 (3)	1.500 (7)
C(2)—C(3)	1.520 (5)	1.526 (4)	1.533 (7)
C(3)—C(4)	1.513 (5)	1.521 (5)	1.534 (7)
C(4)—C(5)	1.506 (4)	1.509 (4)	1.527 (8)
C(5)—C(6)	1.524 (5)	1.516 (4)	1.528 (8)
C(6)—C(1)	1.499 (4)	1.509 (4)	1.500 (7)
C(4)—C(8)			1.538 (7)
C(8)—C(9)			1.539 (10)
C(8)—C(10)			1.543 (10)
C(8)—C(11)			1.522 (12)
O(1)—C(7)—N(3)	123.1 (2)	122.4 (2)	122.3 (5)
O(1)—C(7)—N(2)	121.1 (2)	120.6 (2)	120.6 (4)
N(3)—C(7)—N(2)	115.8 (2)	116.9 (2)	117.0 (5)
N(1)—N(2)—C(7)	117.7 (2)	117.1 (2)	115.7 (4)
N(2)—N(1)—C(1)	119.7 (2)	119.6 (2)	119.5 (4)
N(1)—C(1)—C(2)	127.5 (2)	128.6 (2)	129.4 (4)
N(1)—C(1)—C(6)	116.8 (2)	115.6 (2)	117.1 (4)
C(2)—C(1)—C(6)	115.7 (2)	115.7 (2)	113.6 (4)
C(1)—C(2)—C(3)	110.9 (3)	111.9 (2)	111.0 (4)
C(2)—C(3)—C(4)	112.4 (2)	112.7 (2)	113.5 (5)
C(3)—C(4)—C(5)	109.8 (2)	111.0 (2)	108.2 (5)
C(4)—C(5)—C(6)	111.3 (3)	111.1 (3)	110.7 (5)
C(5)—C(6)—C(1)	112.1 (2)	111.7 (2)	111.1 (4)
C(3)—C(4)—C(8)			114.6 (5)
C(5)—C(4)—C(8)			114.5 (5)
C(4)—C(8)—C(9)			109.8 (5)
C(4)—C(8)—C(10)			109.1 (5)
C(4)—C(8)—C(11)			112.3 (6)
C(9)—C(8)—C(10)			106.3 (6)
C(9)—C(8)—C(11)			108.8 (6)
C(10)—C(8)—C(11)			110.3 (6)

For both compounds data were corrected for Lorentz and polarization effects but not for absorption. The structures were determined by direct methods with *SIR*88 (Burla *et al.*, 1989) and refined by anisotropic full-matrix least squares. The H atoms were localized from final difference Fourier syntheses and refined isotropically. For BCSC the isotropic temperature factor of the H atoms was arbitrarily fixed to the corresponding value of the attached non-H atoms. There are no significant differences between the two crystallographically independent molecules of CSC. Calculations were carried out on a Data General Eclipse MV/8000 II using mainly *SIRCAOS* (Camalli *et al.*, 1986). Some of the final calculations were performed with *PARST* (Nardelli, 1983). The scattering factors used were those of Cromer & Mann (1968) for the non-H atoms and those of Hanson, Herman, Lea & Skillman (1964) for the H atoms.

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1,1';2,2'-Bis-μ-dimethylsilyl-bis[1,2-dicarba-*closo*-dodecaborane(12)], $C_8H_{32}B_{20}Si_2$

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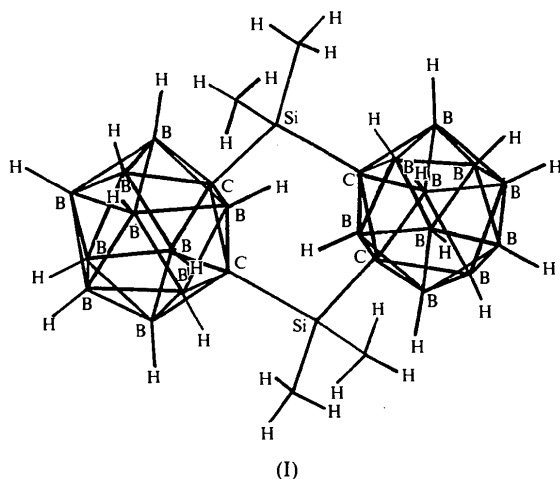
Abstract

The molecule is situated across the center of inversion. Two *closo*-carbaborane cages are joined by two Si bridges with Si—C bonds of 1.907 (3) and 1.914 (3) Å.

Comment

Our research on the coordination chemistry of macrocyclic derivatives of S,S'-connected strings of the moiety 7,8-dimercapto-7,8-dicarba-*nido*-undecaborate(1−) (Teixidor, Viñas, Rius, Miravitlles & Casabó, 1990, and references therein; Teixidor, Casabó, Viñas, Sanchez, Escriche & Kivekäs, 1991) has shown the singularity of their chemistry. These studies have been extended to monomercapto-*nido*-carbaborane species, which have shown remarkable behavior. One example is the synthesis of PdPPh$_3$Cl-{7-SMe-8-Me-11-PPh$_2$-7,8-C$_2$B$_9$H$_{10}$}, where a P—C and a B—H bond have been broken and a P—B bond has been formed under mild conditions (Teixidor, Casabó, Romerosa, Viñas, Rius & Miravitlles, 1991). To obtain further insight into these monomercapto-*nido*-carbaborane species, we attempted to synthesize 2,2'-μ-dimethylsilyl-bis-(1-mercapto-1,2-dicarba-*closo*-dodecaborane) by the reaction of (CH$_3$)$_2$SiCl$_2$ with lithium 1,2-dicarba-*closo*-dodecaborate(1−) (molar ratio 1:2) in dry benzene, followed by ulterior lithiation and nucleophilic attack to elemental sulfur to produce the desired thiol compound. This reaction did not proceed as expected but yielded a solid which produced good-quality crystals of 1,1';2,2'-bis-μ-dimethylsilyl-bis(1,2-dicarba-*closo*-dodecaborane), (I), which contain a six-membered ring containing two *ortho*-carbaborane units. The preparation of this compound has been reported previously by Papetti and co-workers (Papetti & Heying, 1963; Papetti, Schaeffer, Troscianiec & Heying, 1964). Here we report its crystal structure.

(I)

The molecule is situated across a crystallographic center of inversion but the approximate symmetry is *mmm*. The molecule consists of two *closo*-carbaborane cages joined by two Si bridges. The angles around the Si atom (108.4–111.9°) are near the ideal tetrahedral value, indicating the absence of

strain at Si. Comparison of the bond parameters around the Si atom in the title compound with those of 1,1';2,2'-bis-μ-dichlorosilyl-bis(1,2-dicarba-*closo*-borane) (Rayn & Schaeffer, 1981) gave slight differences. The C7—Si—C8^i angle of 111.0 (2)° is smaller in the title compound than that in the corresponding dichloro compound [114.9 (1)°], but the Si—C(carbaborane) distances of 1.907 (3) and 1.914 (3) Å are slightly longer in the title compound than those in the dichloro compound [1.880 (3) and 1.881 (3) Å]. The deviation of *ca* 0.03 Å may be attributed to the larger electronegativity of the Cl atom *versus* the C atom, which makes the Si atom more positive in the Cl derivative. Consequently, the C(carbaborane)—Si(Cl) bond has a larger ionic contribution than C(carbaborane)—Si(C), thus producing a comparatively shorter distance. The bond distances and angles of the carbaborane cage do not deviate significantly from the expected values.
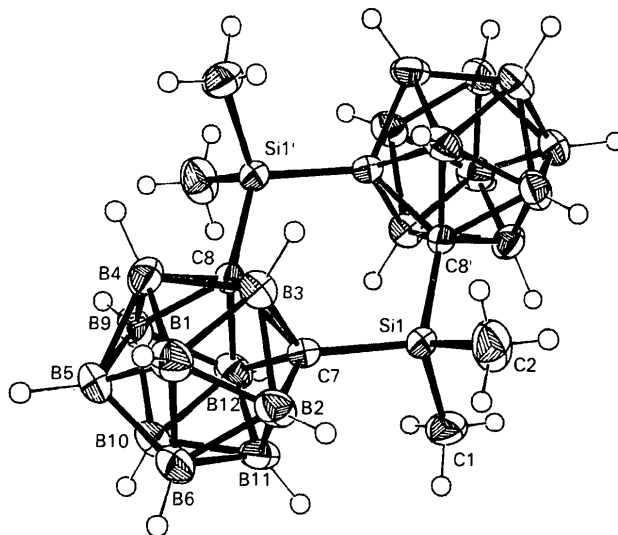

Fig. 1. View of C$_8$H$_{32}$B$_{20}$Si$_2$ showing the labeling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radii.

Experimental

Crystal data

C$_8$H$_{32}$B$_{20}$Si$_2$	Mo $K\alpha$ radiation
M_r = 400.74	λ = 0.71069 Å
Monoclinic	Cell parameters from 25
$P2_1/c$	reflections
a = 9.794 (2) Å	θ = 10–14°
b = 9.614 (2) Å	μ = 0.147 mm^{-1}
c = 13.105 (2) Å	T = 295 K
β = 107.74 (2)°	Prism
V = 1175.3 (4) Å^3	0.4 × 0.35 × 0.3 mm
Z = 2	Colorless
D_x = 1.132 Mg m^{-3}	

Data collection

Syntex $P2_1$ diffractometer	$R_{int} = 0.032$
ω scans	$\theta_{max} = 26.5°$
Absorption correction:	$h = 0 \rightarrow 12$
not applied	$k = 0 \rightarrow 13$
($T_{min} = 0.97$, $T_{max} = 1.00$)	$l = -16 \rightarrow 16$
2727 measured reflections	3 standard reflections
2433 independent reflections	monitored every 100
1563 observed reflections	reflections
[$|F| > 4\sigma(F)$]	intensity variation: 2%

Refinement

Refinement on F	$w = 1/\sigma^2(F)$
$R = 0.062$	$(\Delta/\sigma)_{max} = 0.098$
$wR = 0.053$	$\Delta\rho_{max} = 0.31$ e Å^{-3}
$S = 2.64$	$\Delta\rho_{min} = 0.25$ e Å^{-3}
1563 reflections	Extinction correction: none
201 parameters	Atomic scattering factors
All H-atom parameters	from *Xtal*2.6 (Hall &
refined	Stewart, 1989)

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

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

	x	y	z	U_{eq}
Si1	0.1173 (1)	0.1432 (1)	−0.03306 (8)	0.0303 (5)
C1	0.2176 (5)	0.1231 (7)	−0.1314 (4)	0.055 (3)
C2	0.1444 (6)	0.3170 (5)	0.0302 (5)	0.057 (3)
B1	0.2904 (5)	−0.0456 (5)	0.2955 (4)	0.041 (3)
B2	0.3156 (4)	0.0563 (5)	0.1914 (4)	0.038 (2)
B3	0.1392 (5)	0.0168 (5)	0.1931 (3)	0.032 (2)
B4	0.1432 (5)	−0.1563 (5)	0.2395 (4)	0.037 (2)
B5	0.3185 (5)	−0.2216 (5)	0.2666 (4)	0.042 (2)
B6	0.4242 (5)	−0.0903 (5)	0.2346 (4)	0.041 (3)
C7	0.1847 (3)	0.0064 (4)	0.0760 (2)	0.027 (2)
C8	0.0829 (3)	−0.1196 (3)	0.1056 (3)	0.027 (2)
B9	0.1831 (5)	−0.2674 (5)	0.1464 (4)	0.040 (2)
B10	0.3573 (5)	−0.2278 (5)	0.1414 (3)	0.040 (2)
B11	0.3556 (5)	−0.0553 (5)	0.0967 (4)	0.038 (2)
B12	0.2046 (4)	−0.1646 (5)	0.0399 (3)	0.037 (2)

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

Si1—C1	1.853 (6)	C7—B11	1.718 (6)
Si1—C2	1.847 (5)	C7—B12	1.738 (6)
Si1—C7	1.907 (3)	C8—B3	1.718 (5)
Si1—C8^i	1.914 (3)	C8—B4	1.709 (5)
C7—B2	1.726 (5)	C8—B9	1.717 (6)
C7—B3	1.725 (6)	C8—B12	1.725 (6)
C7—C8	1.688 (5)		
Si1—C7—B2	117.4 (2)	Si1^i—C8—B3	119.6 (2)
Si1—C7—B3	120.2 (2)	Si1^i—C8—B4	117.8 (3)
Si1—C7—C8	125.0 (2)	Si1^i—C8—C7	124.0 (2)
Si1—C7—B11	117.0 (3)	Si1^i—C8—B9	117.3 (2)
Si1—C7—B12	119.4 (2)	Si1^i—C8—B12	118.8 (2)
B3—C7—C8	60.4 (2)	B3—C8—C7	60.9 (2)
C8—C7—B12	60.4 (2)	C7—C8—B12	61.2 (2)

Symmetry code: (i) $-x, -y, -z$.

Data were neither corrected for the intensity variation in the standard reflection nor for the minor variation caused by absorption. Cell refinement, data collection and reduction: Syntex $P2_1$. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1986). Program(s) used to refine structure: *Xtal*2.6 (Hall & Stewart, 1989). Molecular graphics: *Xtal*, ORTEP (Johnson, 1976). Software used to prepare material for publication: *Xtal* BONDLA and ATABLE.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71631 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1091]

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Structural Studies on the Antimuscarinic Agents Spiro-DAMP and Hydroxy-DAMP, and Comparison with Related Compounds

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Abstract

Compound (I), 8,8-dimethyl-3,3-diphenyl-1,4-dioxa-8-azoniaspiro[4.5]decan-2-one iodide, spiro-DAMP, has the ester moiety enclosed in a rigid dioxolane ring, giving rise to a spiro piperidine derivative. Compound (II), 4-(2-hydroxy-2,2-diphenylacetoxy)-1,1-dimethylpiperidinium iodide, hydroxy-DAMP, shows the ester substituent in an equatorial position