

O1—C11—O2	109.6 (1)	O2—C11—O4	109.4 (1)
O1—C11—O3	109.9 (1)	O3—C11—O4	109.7 (1)
O1—C11—O4	109.2 (1)	C6—N1—C2	122.5 (2)
O2—C11—O3	109.1 (1)		

Table 2. Hydrogen-bonding geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O1	0.81 (5)	2.22 (4)	2.893 (3)	140 (3)
N1—H1...O2 <sup>i</sup>	0.81 (5)	2.54 (4)	2.915 (3)	110 (3)
N1—H1...O2 <sup>ii</sup>	0.81 (5)	2.36 (4)	2.972 (3)	132 (3)
C3—H3...O4 <sup>iii</sup>	0.94 (3)	2.57 (3)	3.483 (3)	165 (2)
C5—H5...O4 <sup>iv</sup>	0.91 (4)	2.44 (4)	3.343 (3)	172 (3)
C6—H6...O3 <sup>v</sup>	0.97 (3)	2.52 (3)	3.227 (3)	130 (2)

Symmetry codes: (i)  $x-1, y, z$ ; (ii)  $-x, 1-y, 1-z$ ; (iii)  $x-\frac{1}{2}, \frac{1}{2}-y, z-\frac{3}{2}$ ; (iv)  $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$ .

Data collection: *CAD-4/PC* (Kretschmar, 1994). Cell refinement: *CELDIM* (Enraf–Nonius, 1988). Data reduction: *XCAD4* (Harms, 1997). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL97*.

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## 3,3,6,6-Tetramethyl-1-trimethylsilyl-3,6-diaza-1,4,5-tricarbaheptaborane(10)†

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## Abstract

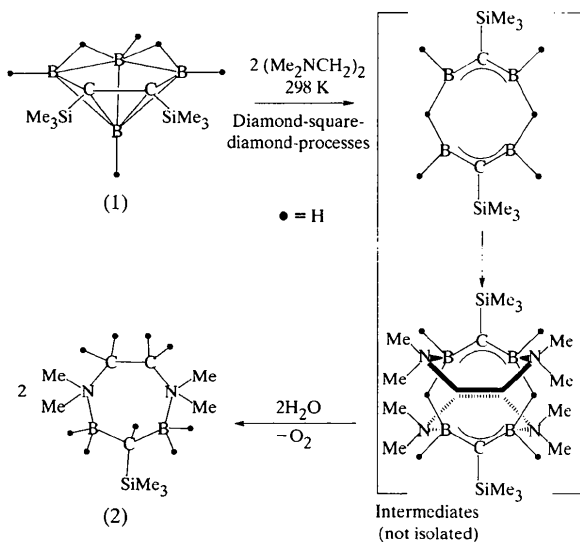
The title compound, C<sub>10</sub>H<sub>30</sub>B<sub>2</sub>N<sub>2</sub>Si, was synthesized in 98% yield. It has a zwitterionic heterocyclic carborane ring in which an *N,N,N',N'*-tetramethylethylenediamine (TMEDA) unit is coordinated *via* two donor N atoms to two BH<sub>2</sub> units that are separated by a CH(SiMe<sub>3</sub>) moiety to form a novel seven-membered ring. The bond distances (Å) within the ring are B—C 1.613 (3) and 1.618 (3), B—N 1.652 (3) and 1.659 (3), C—N 1.483 (3) and 1.505 (3), and C—C 1.510 (3).

## Comment

The dominant structural patterns found in alkyl and aromatic hydrocarbons are reflections of the structures of elemental carbon (diamond or graphite), where an added H atom at the end of C—C bonds gives 'electron-precise carbon hydrides' (Williams, 1998). In a similar way, the structures of boron hydrides are reflections of the icosahedral units found in elemental boron and the replacement of a BH unit by a CH or CR unit gives the corresponding 'electron-deficient' carborane derivatives (Lipscomb, 1963; Muetterties & Knoth, 1968; Parry, 1998). Although our recent communication (Hosmane *et al.*, 1996) reported the first example of a polyhedral cluster that consisted of both electron-precise atoms and an electron-deficient carborane unit in a single cage framework, there has been no report on the conversion of an electron-deficient carborane cage essentially into an electron-precise heterocyclic ring. As part of an exploration of new methodologies in this area, the title compound, 3,6-dimethyl-1-trimethylsilyl-3,6-diaza-1,4,5-tricarbaheptaborane(10), (2), was synthesized. We report here the results of this investigation where

† Alternative name: 1,1,5,5-tetramethyl-3-trimethylsilyl-1,5-diazonia-2,4-diboroniacycloheptane.

an 'electron-deficient' polyhedral cage undergoes transformation to yield the corresponding seven-membered 'electron-precise' heterocyclic ring.



The exclusive formation of (2) from (1) clearly indicates that the transformation is real and is essentially quantitative. Although the exact mechanism is not known, a plausible sequence for the transformation could be that the presence of two lone pairs of electrons from the donor N atoms of *N,N,N',N'*-tetramethylethylenediamine (TMEDA) initiates the reductive cage-opening process, as in the synthesis of a 'carbons apart' *nido*-carborane cage from its corresponding *closo*-carborane precursor (Hosmane *et al.*, 1991; Zhang *et al.*, 1993). The repetition of the process forms a six-membered electron-delocalized carborane ring whose 1,6-positions are occupied by C—SiMe<sub>3</sub> units. The formation of a six-membered ring from a carborane cage can be viewed as the result of several diamond-square-diamond (DSD) processes similar to those observed in the conversion of *ortho*- to *meta*- to *para*-carborane (Lipscomb & Britton, 1960; Lipscomb, 1966). The B atoms in the resulting hexagonal ring can bond to N atoms of two separate TMEDA molecules so as to form two identical seven-membered rings connected by two B—H—B<sub>bridge</sub> bonds. The subsequent redox reaction of this species with water molecules could produce the title compound (2).

The crystal structure (Fig. 1) reveals that (2) is a novel zwitterionic 'electron-precise' heterocyclic seven-membered ring whose bond distances are such that the ring atoms all form single bonds (Table 1). Within experimental error, all of the bond angles within the ring indicate a distorted tetrahedral arrangement for each ring atom. Consequently, the positive charge on the N atoms are balanced by the negative charge on the B atoms, resulting in a zwitterionic heterocyclic ring

in (2). Furthermore, because of the electron-deficient nature of the B atoms on both sides, coordination about C is flattened for a Csp<sup>3</sup> atom. To our knowledge, this compound constitutes the first example of a heterocyclic ring that is not only zwitterionic, but which comprises atoms of groups 13, 14 and 15 in a single ring.

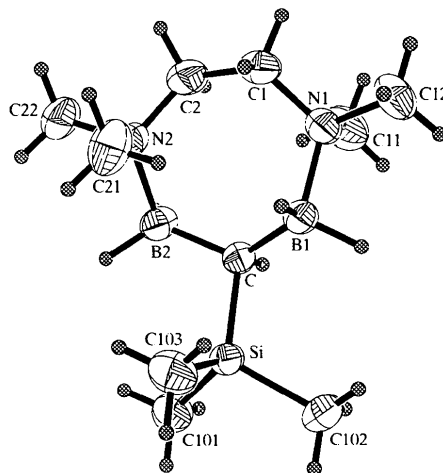


Fig. 1. Displacement-ellipsoid drawing of the title compound shown at the 50% probability level.

This work together with our recent results on carborane cage-opening reactions to yield the corresponding 'carbons apart' carborane isomers indicated that caution must be exercised during the synthesis of metallocarboranes involving strong Lewis bases, such as TMEDA. Nonetheless, the significant role of TMEDA in the cage opening of polyhedral *nido*-carboranes is herein exemplified.

## Experimental

A neat sample of *nido*-2,3-(SiMe<sub>3</sub>)<sub>2</sub>-2,3-C<sub>2</sub>B<sub>4</sub>H<sub>6</sub> [(1); 1.10 g, 5.00 mmol] was reacted with a large excess of wet TMEDA *in vacuo* with constant stirring at room temperature for 7 d, producing a colorless crystalline solid (1.12 g, 98% yield). The <sup>1</sup>H, <sup>11</sup>B and <sup>13</sup>C NMR spectra while showing the absence of a carborane cage were not sufficient to identify the product unambiguously (Hosmane, 1999).

### Crystal data

C<sub>10</sub>H<sub>30</sub>B<sub>2</sub>N<sub>2</sub>Si  
M<sub>r</sub> = 228.07  
Orthorhombic  
P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
a = 6.4731 (1) Å  
b = 11.4104 (1) Å  
c = 20.4092 (3) Å  
V = 1507.44 (3) Å<sup>3</sup>  
Z = 4  
D<sub>x</sub> = 1.005 Mg m<sup>-3</sup>  
D<sub>m</sub> not measured

Mo Kα radiation  
λ = 0.71073 Å  
Cell parameters from 130 reflections  
θ = -11.5–11.5°  
μ = 0.132 mm<sup>-1</sup>  
T = 293 (2) K  
Fragment  
0.6 × 0.3 × 0.3 mm  
Light yellow

**Data collection**

Siemens SMART CCD  
PLATFORM diffractometer  
 $\omega$  scans  
Absorption correction:  
multi-scan (SADABS;  
Sheldrick, 1996)  
 $T_{\min} = 0.749$ ,  $T_{\max} = 0.924$   
9854 measured reflections  
2084 independent reflections  
(plus 1470 Friedel-related reflections)

3384 reflections with  
 $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.024$   
 $\theta_{\text{max}} = 27.86^\circ$   
 $h = -6 \rightarrow 8$   
 $k = -14 \rightarrow 14$   
 $l = -25 \rightarrow 26$   
Intensity decay: none

**Refinement**

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.043$   
 $wR(F^2) = 0.115$   
 $S = 1.243$   
3554 reflections  
157 parameters  
H atoms treated by a  
mixture of independent  
and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.2776P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.019$

$\Delta\rho_{\text{max}} = 0.21 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{\AA}^{-3}$   
Extinction correction:  
SHELXL (Sheldrick, 1997)  
Extinction coefficient:  
0.0009 (18)  
Scattering factors from  
International Tables for  
Crystallography (Vol. C)  
Absolute structure:  
Flack (1983)  
Flack parameter =  
-0.07 (14)

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Si—C	1.8551 (18)	B2—C	1.618 (3)
Si—C101	1.878 (2)	B2—N2	1.659 (3)
Si—C102	1.877 (2)	C1—N1	1.483 (3)
Si—C103	1.877 (2)	C1—C2	1.510 (3)
B1—C	1.613 (3)	C2—N2	1.505 (3)
B1—N1	1.652 (3)		
C—Si—C101	112.20 (10)	B2—C—Si	109.94 (13)
C—Si—C102	112.29 (10)	N1—C1—C2	118.62 (19)
C101—Si—C102	104.68 (11)	N2—C2—C1	118.92 (19)
C—Si—C103	111.96 (10)	C11—N1—C1	111.4 (2)
C101—Si—C103	107.88 (12)	C11—N1—C12	108.0 (2)
C102—Si—C103	107.42 (12)	C1—N1—C12	105.92 (18)
C—B1—N1	114.01 (14)	C11—N1—B1	110.82 (17)
C—B2—N2	115.93 (15)	C1—N1—B1	113.02 (16)
B1—C—B2	125.86 (15)	C12—N1—B1	107.39 (16)
B1—C—Si	106.54 (12)		

H atoms on C, B1 and B2 were located by difference map and were refined individually. Other H atoms were refined as riding, with  $U_{\text{iso}}$  values 1.3 times those of the parent atoms, and C—H distances of 0.97  $\text{\AA}$  for  $\text{CH}_2$  and 0.96  $\text{\AA}$  for  $\text{CH}_3$  groups.

Data collection: SMART (Siemens, 1995). Cell refinement: SMART and SAINT (Siemens, 1995). Data reduction: SAINT. Program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999). Program(s) used to refine structure: SHELXL (Sheldrick, 1997). Molecular graphics: SHELXL. Software used to prepare material for publication: SHELXL.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1209). Services for accessing these data are described at the back of the journal.

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**4,6-Diethoxy-1,3,5-triazin-2(1H)-one**

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**Abstract**

The molecules of the title compound,  $\text{C}_7\text{H}_{11}\text{N}_3\text{O}_3$ , are almost planar owing to an extensive conjugation of  $\text{C}=\text{N}$  and  $\text{C}=\text{O}$  double bonds resulting from five purely  $sp^2$  hybridized atoms and one amide-N atom in the ring. The strong hydrogen  $\text{N}-\text{H} \cdots \text{O}=\text{C}$  bonds produce a dimer across a centre of symmetry. The dimers are interconnected by weaker hydrogen bonds to form a layer structure.