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A New Series of Organoboranes IV. The Participation of the 11,12-Dicarbadodecaborane Nucleus in Some Novel Heteratomic Ring Systems

by

Stelvio Papetti and T. L. Heying

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Olin Mathieson Chemical Corporation
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A New Series of Organoboranes IV.
The Participation of the 11,12-Dicarbadodecaborane Nucleus in Some Novel Heteratomic Ring Systems

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Stelvio Papetti and T. L. Heying

Received............

Various chlorosilylcarborane[1] derivatives were prepared. In subsequent reactions with amines or water a marked propensity for the formation of five membered disila azane and disila oxane rings was exhibited. In reactions with dilithiocarborane, novel disilacyclohexane analogs were formed.

In the preceding paper[2] several examples of the synthesis

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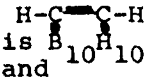
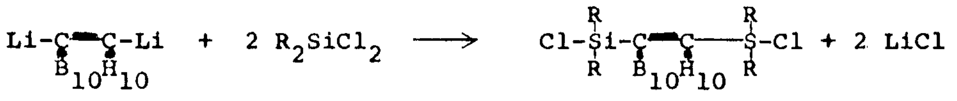
(1) For brevity the term carborane is used in place of the formal name 11,12-dicarbadodecaborane for the compound  $H-C \overset{}{-} C-H$ ($B_{10}H_{10}$). For structure refer to paper I of this series by H. Schroeder, T. L. Heying and J. R. Reiner, Inorg. Chem. 2, in press.

(2) T. L. Heying, J. W. Ager, S. L. Clark, R. P. Alexander, S. Papetti, J. A. Reid and S. I. Trotz, Inorg. Chem. ________.

of mono and disilylcarboranes were given. Utilization of the same general preparative procedures has proven quite satisfactory for the preparation chlorosilylcarboranes in good yield as indicated in reaction 1.

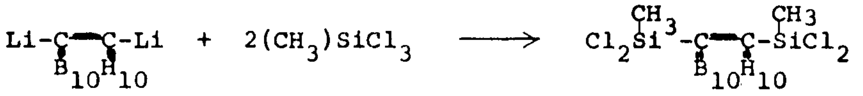


$$\mathrm{Li{-}\underset{B_{10}H_{10}}{C{-}C}{-}Li} + 2\,\mathrm{R_2SiCl_2} \longrightarrow \mathrm{Cl{-}\overset{R}{\underset{R}{Si}}{-}\underset{B_{10}H_{10}}{C{-}C}{-}\overset{R}{\underset{R}{Si}}{-}Cl} + 2\,\mathrm{LiCl} \qquad 1.$$

I - R= $-CH_3$

II - R= $-C_6H_5$

By applying the proper stoichiometry we were able to prepare the corresponding tetrachloro and hexachloro derivatives in surprisingly good yield.



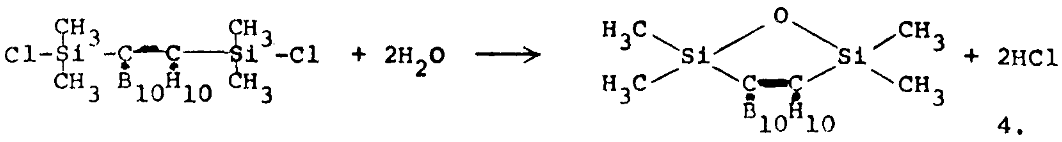
$$\mathrm{Li{-}\underset{B_{10}H_{10}}{C{-}C}{-}Li} + 2(\mathrm{CH_3})\mathrm{SiCl_3} \longrightarrow \mathrm{Cl_2\overset{CH_3}{Si}{-}\underset{B_{10}H_{10}}{C{-}C}{-}\overset{CH_3}{Si}Cl_2} \qquad 2.$$

III

$$\mathrm{Li{-}\underset{B_{10}H_{10}}{C{-}C}{-}Li} + 2\,\mathrm{SiCl_4} \longrightarrow \mathrm{Cl_3Si{-}\underset{B_{10}H_{10}}{C{-}C}{-}SiCl_3} \qquad 3.$$

IV

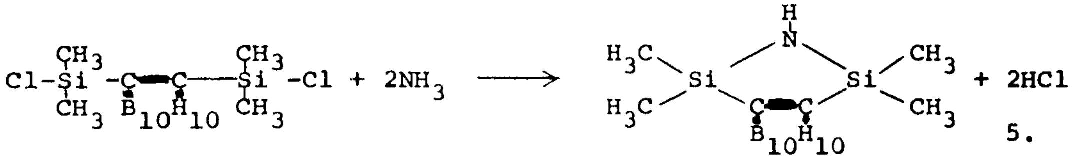
To determine whether bis(chloro dimethylsilyl)carborane (I) would behave chemically as do the conventional organic derivatives, it was subjected to hydrolysis expecting to form a macrocyclic siloxane, a linear silicone or possibly the corresponding dihydroxy compound. To the contrary, under all of the many sets of conditions attempted to date, I was converted to the cyclic tetramethyldisila oxane, V. In most instances it is achieved in high purity in essentially quantitative yield.



V

The cyclic structure was first assigned on the basis of its elemental analysis, molecular weight and infrared spectrum. This assignment was borne out subsequently by examination of its mass spectrum which is in agreement in every detail. The same evidence has been gained for those novel cyclic structures discussed below. Compound V above has also been prepared by the reaction of dilithiocarborane with excess tetramethyl dichlorodisiloxane.

When bis(chloro dimethylsilyl)carborane was treated with ammonia, a similar reaction occurred to give in this instance the cyclic tetramethyl disila azane, VI.

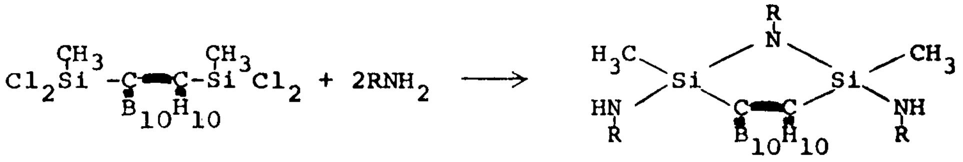


5.

VI

Unlike the numerous organic silicon-nitrogen bonded compounds which have been noted to readily react with moisture, we have been unable to hydrolyze this and related compounds. Also this compound and its disila oxane analog V were recovered quantitatively unchanged after heating at 500°C. Above this temperature some methane was evolved.

Amminolysis of bis(dichloro methylsilyl)carborane with ammonia and methylamine (III) was next studied and here high yields of substituted cyclic disila azanes VI and VII formed.



6.

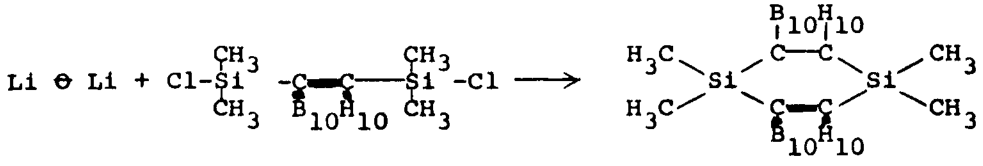
VI - R= -H

VII - R= $-CH_3$

Hydrolysis of these compounds also left the disila azane ring in tact but the pendant -NHR groups were attacked.

The ease with which these reactions occur to form five membered rings bears notice. We have observed a similar situation in other carborane derivatives wherein the three ring atoms other than the carboranyl carbon pair are -C-C-C-, -C-O-C- and -C-Si-C-[2]. Other workers in these laboratories[3] have encountered a similar situation wherein the non-carboranyl ring atoms are -P-N-P-. This suggests that the exonuclear bond angles and the bond distance of the two carborane carbon atoms are of a magnitude particularly suited for participation in five membered rings. Also, to our knowledge, there is no instance of the participation of one carboranyl nucleus in a larger ring and this further suggests that this bond angle is quite invariant. Further support of this concept comes from the fact that the infrared carbon-hydrogen stretching absorption occurs at 3.25μ[1] which is essentially the same as that of the rigidly oriented acetylenic ≡C-H.

When bis(chloro dimethylsilyl)carborane was treated with dilithiocarborane a reaction occurred which gave a product having a six membered ring composed of four carbon atoms of two carborane nuclei and two silicon atoms.



7.

VIII

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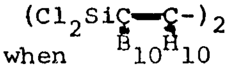
(3) R. P. Alexander and H. Schroeder, Inorg. Chem. ______

All chemical and instrumental analyses are in complete agreement again. This too seems to be a favored type of ring system since an analogous system wherein the silicon atoms are replaced by phosphorus III has recently been discovered[3]. When bis(trichlorosilyl)carborane was similarly treated with an equimolar quantity of dilithiocarborane the analogous reaction occurred to give the cyclic tetrachloroderivative  $(Cl_2Si\underset{B_{10}H_{10}}{C{-}C}-)_2$ (X). It is of interest that no change in X occurred when it was treated with water at room temperature thereby not showing the normal tendency of the silicon-chlorine for hydrolysis. This phenomenon is being investigated in more detail as is the general preparative and polymer chemistry of all of the novel heterocyclic compounds herein reported.

EXPERIMENTAL

Bis(chloro dimethylsilyl) carborane I: A dilithiocarborane slurry[2] was prepared from 50.0 gr. (0.347 mol.) carborane[4] and 2.05 moles of butyl lithium in ether and slowly added to a stirred, etheral solution of 98.33 gr. (0.762 mol.) dichloro dimethylsilane cooled in an ice bath. When addition was complete the mixture was refluxed overnight and then filtered. The filtrate was evaporated to dryness and the residue was sublimed under vacuum at a bath temperature of 125-130°C and the sublimate was recrystallized from heptane. Bis(chloro dimethylsilyl)carborane (m.p. 112.5-113.5°C)[5] was recovered in 88% yield.

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(4) We thank Mr. R. W. Blundon for preparing a continuing supply of carborane for this work.

(5) All melting points were taken on Mel-Temp Apparatus and are uncorrected.

Anal:[6] Calc'd for $C_6H_{22}B_{10}Cl_2Si_2$: C, 21.84; H, 6.86; B, 32.79; Cl, 21.49; Si, 17.02

Found: C, 22.05; H, 6.67; B, 32.57; Cl, 21.15; Si, 16.79

Bis(chloro diphenylsilyl)carborane II: The reaction was identical with that for I using 15.35 gr. (0.106 mol.) of carborane and 59.3 gr. (0.234 mol.) of dichloro diphenylsilane. The sublimation was omitted and the 22.7% yield of bis(chloro diphenylsilyl)carborane (m.p. 244-245°C) was obtained directly by recrystallization from heptane.

Anal: Calc'd for $C_{26}H_{30}B_{10}Cl_2Si_2$: C, 54.05; H, 5.23; B, 18.12; Cl, 12.26; Si, 9.73

Found: C, 53.80; H, 5.13; B, 18.80; Cl, 12.15; Si, 9.27

Bis(dichloro methylsilyl)carborane III: This compound was prepared as was II but from 21.6 gr. (0.150 mol.) of carborane and 49.7 gr. (0.333 mol.) of methyl trichlorosilane. The 69.5% yield of bis(chloro methylsilyl)carborane (m.p. 119-120°C) was recovered from recrystallization from 30-60° petroleum ether.

Anal: Calc'd for $C_4H_{16}B_{10}Cl_4Si_2$: C, 12.97; H, 4.35; B, 29.22; Cl, 38.29; Si, 15.17

Found: C, 13.10; H, 4.34; B, 29.30; Cl, 38.35; Si, 14.75

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(6) Analyses were by the Olin Microanalytical Section.

Bis(trichlorosilyl)carborane IV: The procedure of II was followed using 15.87 gr. (0.11 mol.) carborane and 41.15 gr. (0.242 mol.) of tetrachlorosilane. The crude product was recrystallized from heptane to give a 60% yield of bis(trichlorosilyl)carborane (m.p. 121-122°C).

Anal: Calc'd for $C_2H_{10}B_{10}Cl_6Si_2$: C, 5.85; H, 2.43; B, 26.31; Cl, 51.74; Si, 13.67

Found: C, 5.74; H, 2.51; B, 26.61; Cl, 51.35; Si, 13.17

C,C'(1,1,3,3-tetramethyldisila oxanyl-1,3)carborane V: This compound is readily prepared by adding excess water to a benzene-acetone solution of bis(chloro dimethylsilyl)carborane (I) at room temperature. The resulting solution is concentrated under reduced pressure until most of benzene and acetone are removed, the remaining mixture filtered and the residue is either recrystallized from heptane or sublimed. A practically quantitative yield of V (m.p. 160-161°C) is obtained.

Anal: Calc'd for $C_6H_{22}B_{10}OSi_2$: C, 26.20; H, 8.07; B, 39.33; Si, 20.42

Found: C, 26.55; H, 8.06; B, 39.30; Si, 19.7

This compound (V) was prepared in 74.8% yield when dilithiocarborane was reacted with excess tetramethyl dichlorodisiloxane.

C,C'(1,1,3,3-tetramethyldisila azanyl-1,3)carborane VI:

Bis(chloro dimethylsilyl)carborane (I) (2.69 gr., .0081 mol.) was dissolved in 50 ml. of ethyl ether and cooled in an ice bath. Ammonia was passed through at a rapid rate for 15 minutes during which time a precipitate formed. The mixture was filtered and the filtrate was evaporated to dryness and the residue so formed was recrystallized from high boiling petroleum ether to give an essentially quantitative yield of VI (m.p. 190-192°C).

Anal: Calc'd for $C_6H_{23}B_{10}NSi_2$: C, 26.29; H, 8.46; B, 39.49;
N, 5.11; Si, 20.49; M.W., 274

Found: C, 26.58; H, 8.21; B, 39.70;
N, 5.05; Si, 19.6; M.W., 270
(osmometric)

<u>C,C'(1,3-diamino-1,3-dimethyldisila azanyl-1,3)carborane</u> VII:

The procedure for VI was followed exactly using 2.0 gr. (.006 mol.) of bis(dichloro methylsilyl)carborane (III) to give a quantitative yield of VII (m.p. 189-191.5°C).

Anal: Calc'd for $C_4H_{21}B_{10}NSi_2$: C, 17.42; H, 7.68; B, 39.26;
N, 15.25; Si, 20.38; M.W., 276

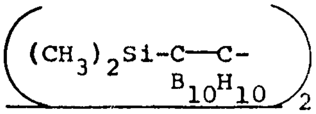
Found: C, 17.41; H, 7.83; B, 38.48;
N, 14.64; Si, 20.08; M.W., 278
(cryoscopic)

<u>C,C'(1,2,3-trimethyl-1,3-dimethylaminodisila azanyl-1,3)carborane</u> VIII:

Methylamine was bubbled through a cooled solution of 4.7 gr. (0.014 mol.) bis(dichloro methylsilyl)carborane (III) in 60 ml. ether for 30 minutes. The mixture was filtered, the filtrate concentrated and the resulting solid was recrystallized from 30-60°C petroleum ether to give an essentially quantitative yield of VIII (m.p. 128-129.5°C).

Anal: Calc'd for $C_7H_{27}B_{10}N_3Si_2$: N, 13.23; Si, 17.68; M.W., 318

Found: N, 13.31; Si, 17.68; M.W., 325
(cryoscopic)

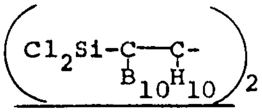


$\left((CH_3)_2Si{-}\underset{B_{10}H_{10}}{C{-}C}{-}\right)_2$

IX: A dilithiocarborane, slurry in ether prepared from 7.5 gr. (0.052 mol.) of carborane was added to a cooled solution of 0.11 mol. of bis(chloro dimethylsilyl)carborane (I) in ether. The mixture was refluxed overnight, filtered and the filtrate evaporated to dryness. This residue was heated at 130-140°C in vacuo to remove unreacted I. The residue was recrystallized from acetone to give a 23% (based on the original amount of I) yield of IX (m.p. 309-310°C).

Anal: Calc'd for $C_8H_{32}B_{20}Si_2$: C, 23.97; H, 8.05; B, 53.97; Si, 14.01; M.W., 400

Found: C, 22.95; H, 7.78; B, 53.84; Si, 14.42; M.W., 379 (osmometric)



$\left(Cl_2Si{-}\underset{B_{10}H_{10}}{C{-}C}{-}\right)_2$

X: Bis(trichlorosilyl)carborane (0.484 gr., 0.0118 mol.) in 50 ml. of ether was allowed to react with dilithiocarborane prepared from 1.550 gr. (0.0107 mol.) of carborane in the usual manner. The reaction was refluxed overnight and then filtered. The residue was extracted several times with ether and the combined extracts were evaporated to dryness. This solid was recrystallized from benzene (2.0 gr.). The filtrate from the original reaction was concentrated and the solid which formed was removed and recrystallized from benzene (0.5 gr.). All mother liquors were combined, evaporated to dryness and this solid was also recrystallized from benzene (0.2 gr.). All three materials were shown to be identical which gave a 52.2% yield of X (m.p. 271-272°C).

Anal: Calc'd for $C_4H_{20}B_{10}Cl_4Si_2$: C, 9.95; H, 4.18; Cl, 29.39

Found: C, 9.74; H, 4.11; Cl, 29.7

Mass spectral analysis also identified the product as X in greater than 98% purity.

ACKNOWLEDGEMENT

The authors thank Mr. Herman Hoberecht for obtaining and interpreting the mass spectra of these compounds which conclusively allowed the assignment of the ring structures and also to Dr. H. Schroeder for his suggestions. This work was sponsored by the Office of Naval Research.

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