Volume 7
Number 3
March 1, 1968

Inorganic Chemistry

CONTRIBUTION NO. 1525 FROM THE DEPARTMENT OF CHEMISTRY,
INDIANA UNIVERSITY, BLOOMINGTON, INDIANA 47401

Studies of Boranes. XXIII. The Chemistry of Isononaborane-15[1]

BY JERRY DOBSON, PHILLIP C. KELLER, AND RILEY SCHAEFFER

Received August 11, 1967

At $-80°$ KB_9H_{14} reacts with anhydrous hydrogen chloride to form a new boron hydride, i-B_9H_{15}, an isomer of the previously reported n-B_9H_{15}. Above $-35°$ i-B_9H_{15} decomposes with elimination of hydrogen to form B_8H_{12}, $B_{10}H_{14}$, n-$B_{18}H_{22}$, and polymeric material. A mechanism is suggested involving initial hydrogen elimination to form B_9H_{13}, which either disproportionates or condenses to generate B_8H_{12} and $B_{10}H_{14}$ or n-$B_{18}H_{22}$, respectively. Treatment of either KB_9H_{14} or $(CH_3)_4N^+B_9H_{12}^-$ in ether with hydrogen chloride at room temperature generates $B_9H_{13}\cdot OR_2$ from which other B_9H_{13}(ligand) complexes may be prepared by ligand displacement. When $B_9H_{13}O(n\text{-}B_4H_9)_2$ is refluxed in $(n\text{-}C_4H_9)_2O$ for 1 hr, n-$B_{18}H_{22}$ is produced in 35% yield.

Introduction

In 1963 Beall and Lipscomb observed that treatment of $(C_2H_5)_3NH^+B_9H_{14}^-$ with polyphosphoric acid produced a mixture of boron hydrides which included B_2H_6, B_4H_{10}, B_5H_9, B_5H_{11}, B_6H_{10}, B_8H_{12}, and $B_{10}H_{14}$.[2] We have found that treatment of KB_9H_{14} with anhydrous hydrogen chloride at low temperature in the absence of electron pair donors produces a new unstable boron hydride, i-B_9H_{15},[3] an isomer of the previously known n-B_9H_{15}.[4] In this paper we present the results of a study of the chemistry of i-B_9H_{15} and its decomposition in the absence of donor molecules.

Experimental Section

High-vacuum techniques used in this investigation have been described elsewhere.[5,6] Vacuum line fractionation was carried out using mercury float valves and O-ring stopcocks (Delmar Scientific Laboratories, Inc.).

All materials used in this work were either reagent grade or were prepared and purified by standard methods. Solvents were dried over $LiAlH_4$ and distilled immediately before use.

The nuclear magnetic resonance spectra were obtained using a Varian Model 4300B spectrometer operating at 19.3 MHz. Low-temperature spectra were obtained with standard Varian accessories.

The mass spectrum of B_8H_{12} was obtained using a Consolidated Electrodynamics Corp. Model 21-620A spectrometer.

Rapid Decomposition of i-B_9H_{15} in the Absence of Solvent.—In a typical experiment 3.00 g (20.0 mmoles) of KB_9H_{14}[7] was converted to i-B_9H_{15} by treatment with excess liquid hydrogen chloride for 30 min at $-78°$ in a 100-ml bulb equipped with a break-off tip. After excess hydrogen chloride had been removed, the product was allowed to warm to $-30°$. At this temperature hydrogen was slowly evolved; the cooling bath was removed and slight warming was allowed until the hydrogen evolution became sufficiently rapid and the rate of reaction was further controlled by cooling with a $-80°$ bath as necessary. The decomposition is evidently somewhat exothermic and during several preparations it appeared that the reaction was accelerating beyond control. Care should be taken during this experiment to have adequate means for cooling at hand at all times. Hydrogen was continually pumped from the system through two traps cooled by liquid nitrogen. Owing to the high thermal instability of the product B_8H_{12}, it was convenient to place the first trap as close as possible to the reaction vessel to minimize decomposition of the substance. The material distilling from the bulb during the decomposition was found to be nearly pure B_8H_{12} containing a small amount of $B_{10}H_{14}$. The B_8H_{12} was separated from the latter by fractionation through a $-30°$ trap. The yield of B_8H_{12} was estimated by measuring the volume of the frozen sample at $-45°$ in a calibrated tube and using the density of the solid as determined by X-ray methods[8] to calculate the weight. In this experiment 0.19 ml, estimated to be 1.7 mmoles, was isolated. The product was identified as B_8H_{12} by its mass spectrum[9] and its hydrolytic hydrogen to boric acid ratio.

The reaction vessel containing the less volatile products of the decomposition of i-B_9H_{15} was opened to the atmosphere and was washed onto a filter with hexane and rinsed several times with the same solvent. Evaporation of the filtrate yielded a sticky

---

[1] For paper XXII of this series see A. Norman and R. Schaeffer, *J. Phys. Chem.*, **70**, 1662 (1966).

[2] H. A. Beall and W. N. Lipscomb, *Inorg. Chem.*, **3**, 1783 (1964).

[3] J. Dobson, P. C. Keller, and R. Schaeffer, *J. Am. Chem. Soc.*, **87**, 3522 (1965).

[4] (a) W. V. Kotlensky and R. Schaeffer, *ibid.*, **80**, 4517 (1958); (b) A. B. Burg and R. Kratzer, *Inorg. Chem.*, **1**, 725 (1962).

[5] A. Stock, "Hydrides of Boron and Silicon," Cornell University Press, Ithaca, N. Y., 1933.

[6] R. T. Sanderson, "High Vacuum Manipulation of Volatile Compounds," John Wiley and Sons, Inc., New York, N. Y., 1948.

[7] L. E. Benjamin, S. F. Stafiej, and E. A. Takacs, *J. Am. Chem. Soc.*, **85**, 2674 (1963). An aqueous solution of $B_9H_{14}^-$ was prepared as described in this reference. The potassium salt was obtained by salting out with K_2CO_3 followed by extraction with diethyl ether.

[8] R. E. Enrione, F. P. Boer, and W. N. Lipscomb, *Inorg. Chem.*, **3**, 1659 (1964).

[9] R. E. Enrione, F. P. Boer, and W. N. Lipscomb, *J. Am. Chem. Soc.*, **86**, 1451 (1964).

yellow residue which was transferred to a vacuum sublimator. The sublimator was slowly warmed to 120°; some hydrogen was evolved and a solid was collected on the cold finger. This solid was first resublimed at room temperature and pure $B_{10}H_{14}$ was thus recovered; subsequently the temperature was raised and at 120° n-$B_{18}H_{22}$ was collected. The $B_{10}H_{14}$ was identified by its volatility and its characteristic ^{11}B nmr spectrum. The n-$B_{18}H_{22}$ was identified by its ^{11}B nmr spectrum,[10] melting point, and ultraviolet spectrum,[11] $B_{10}H_{14}$ recovered, 0.293 g (1.3 mmoles).

Rapid Decomposition of i-B_9H_{15} in Solution.—A 3.00-g (20.0-mmole) sample of KB_9H_{14} was placed in a 500-ml flask with a magnetic stirrer and converted to i-B_9H_{15}, and the excess hydrogen chloride was removed. Approximately 100 ml of dry n-pentane was then condensed onto the residue. The vessel was warmed to −45° and the contents stirred at this temperature for several minutes. The −45° bath was removed and replaced with one at −30° contained in an uninsulated vessel. The temperature of the system was then allowed to rise with constant stirring to about −5° over a 10-min period. The pressure of the evolved hydrogen was kept below 1 atm by allowing it to expand into the vacuum line and partially evacuating when necessary. When the decomposition appeared to be nearly complete, as evidenced by only a slow pressure rise, the flask was cooled to −78°, the cooling bath was removed, and the major portion of the n-pentane was distilled away. The last few milliliters of solvent contained most of the B_8H_{12} and all of the other products. To separate this mixture the pentane was first stripped off at −78°; the trap was then warmed to −30° and the B_8H_{12} was distilled away from the $B_{10}H_{14}$. A small amount of B_8H_{12} was also recovered from the main portion of the pentane solvent by this procedure. Recovered: B_8H_{12}, 0.284 g (2.9 mmoles); $B_{10}H_{14}$, 0.500 g (4.1 mmoles); n-$B_{18}H_{22}$, 0.281 g (1.3 mmoles).

Slow Decomposition of i-B_9H_{15} in Solution.—In a typical reaction 6.30 g (42.0 mmoles) of KB_9H_{14} was converted to i-B_9H_{15} in a 500-ml bulb containing a magnetic stirrer. After removing excess hydrogen chloride, approximately 150 ml of dry toluene was condensed into the bulb. The reaction vessel was placed in a −45° bath and the bath allowed to warm slowly. The solution was stirred continuously and hydrogen was periodically removed. After approximately 6 hr the bath had reached 0° and evolution of hydrogen had nearly ceased. The cold bath was removed and toluene was distilled out as the vessel warmed to room temperature. There appeared to be traces of B_8H_{12} in the last portions of toluene; however, separation of the mixture proved to be impractically difficult since only traces of B_8H_{12} were present. The residue was allowed to stand at room temperature for about 4 hr as slow hydrogen evolution continued. The residue was transferred to a Soxhlet apparatus and extracted with dry n-pentane. The pentane solution was evaporated and the resulting residue was transferred to a high-vacuum sublimator. The $B_{10}H_{14}$ (2.9 mmoles) sublimed at room temperature and 12.2 mmoles of n-$B_{18}H_{22}$ was then collected by sublimation at 120°.

This decomposition was also carried out in a separate experiment on an nmr tube scale. The solution was allowed to warm slowly by gradually raising the temperature of the nmr probe. The nmr signal clearly showed that the principal product of the decomposition was n-$B_{18}H_{22}$. At no time was there an appreciable quantity of B_8H_{12} present.

Preparation of $B_9H_{13}O(C_2H_5)_2$ from KB_9H_{14}.—A 15-mm o.d. reaction tube was charged with an approximately 2-mmole sample of freshly prepared KB_9H_{14}. The vessel was evacuated, and approximately 2 mmoles of hydrogen chloride and 4 ml of dry diethyl ether were condensed into the vessel. The tube was sealed and allowed to come to room temperature. Upon warming, vigorous hydrogen evolution occurred and the nmr spectrum of the resulting solution showed a signal characteristic of B_9H_{13}(ligand) compounds.[12] No attempt was made actually to isolate the etherate of B_9H_{13}. To establish the identity of the above product as a B_9H_{13}(ligand) compound, a 0.931-g (6.21-mmole) sample of KB_9H_{14} was used to form the solution of $B_9H_{13}O(C_2H_5)_2$ as described above. The resulting solution was filtered *in vacuo* onto a 1.567-g (6.0-mmole) sample of $P(C_6H_5)_3$ and the tube was sealed. Upon warming to room temperature the $P(C_6H_5)_3$ dissolved forming a light yellow solution which was allowed to stand at room temperature for 24 hr. During this period large colorless crystals deposited from solution and 1.5 g of crude product was collected by filtration. The compound was then recrystallized from chloroform–hexane. *Anal* for $B_9H_{13}P(C_6H_5)_3 \cdot CHCl_3$.[13] Calcd for $B_9C_{19}H_{29}Cl_3P$: B, 19.7; C, 45.4; H, 5.9; P, 6.3. Found: B, 19.3; C, 45.6; H, 6.3; P, 7.0. A sample of authentic $B_9H_{13}P(C_6H_5)_3$ was prepared as described by Graybill, Pitochelli, and Hawthorne.[12] The product was recrystallized from chloroform–hexane and gave the same powder pattern as the material prepared above.

Preparation of $B_9H_{13}O(C_2H_5)_2$ from $(CH_3)_4N^+B_9H_{12}^-$.—A 15-mm o.d. reaction tube was charged with about 2 mmoles of $(CH_3)_4N^+B_9H_{12}^-$;[14] the vessel was evacuated and an equivalent amount of hydrogen chloride and about 4 ml of diethyl ether were condensed into the vessel. The tube was then sealed and allowed to warm to room temperature. No hydrogen evolution was observed. The ^{11}B nmr spectrum of the solution was identical with the spectrum of $B_9H_{13}O(C_2H_5)_2$ as produced from the reaction of KB_9H_{14} and hydrogen chloride in diethyl ether.

Preparation of n-$B_{18}H_{22}$ by the Pyrolysis of $B_9H_{13}O(n\text{-}C_4H_9)_2$.—A three-neck 500-ml flask was fitted with a pressure-equalizing addition funnel, a reflux condenser, and a mechanical stirrer. The apparatus was charged with 5.81 g (38.7 mmoles) of KB_9H_{14} and 100 ml of dry n-butyl ether, purged with prepurified dry nitrogen, and cooled in an ice bath. A solution of 38.7 mmoles of hydrogen chloride in n-butyl ether was added dropwise with constant stirring over a 30-min period. Hydrogen evolution stopped when the last hydrogen chloride was added to the still colorless solution. The mixture was then refluxed for 1 hr and allowed to cool. The resulting bright yellow solution was extracted several times with 1 N NaOH solution. The aqueous phase was acidified to about pH 1 and extracted with benzene. Evaporation of the benzene solution yielded a yellow residue from which 6.6 mmoles (34% yield) of n-$B_{18}H_{22}$ was recovered by high-vacuum sublimation at 120°. No attempts were made to maximize the yield, and it is possible that a slower decomposition at lower temperatures might have been preferable.

Results and Discussion

The product distribution data for the i-B_9H_{15} decomposition from a number of experiments are summarized in Table I. Rapid decomposition of i-B_9H_{15} with or without solvent appears to be a convenient synthetic route to B_8H_{12}. The high yield obtained from decomposition in solution is somewhat offset by the inconvenience of separating the products from large amounts of pentane and small preparations are conveniently carried out using the solvent-free method. However, in a large preparation (greater than about 20 mmoles of i-B_9H_{15}) the decomposition in pentane is the preferred method not only because of the considerably higher yield but also because the reaction is much more easily controlled. Slow decomposition of i-B_9H_{15} in solution affords excellent yields of n-$B_{18}H_{22}$, although this method suffers from the necessary employment of high-vacuum techniques. The pyrolytic method using

---

[10] W. N. Lipscomb, "Boron Hydrides," W. A. Benjamin, Inc., New York, N. Y., 1963, p 147.

[11] A. R. Pitochelli and M. F. Hawthorne, *J. Am. Chem. Soc.*, **84**, 3218 (1962).

[12] B. M. Graybill, A. R. Pitochelli, and M. F. Hawthorne, *Inorg. Chem.*, **1**, 626 (1962).

[13] Upon pyrolysis of the product *in vacuo*, chloroform was collected and identified by its gas-phase infrared spectrum and vapor pressure.

[14] B. M. Graybill, J. K. Ruff, and M. F. Hawthorne, *J. Am. Chem. Soc.*, **83**, 2669 (1961).

TABLE I

DECOMPOSITION OF i-B_9H_{15} IN PENTANE SOLUTION

		Amount, mmoles				
Expt	Rate	i-B_9H_{15}	n-$B_{18}H_{22}$	$B_{10}H_{14}$	B_8H_{12}	$B_{18}:B_{10}:B_8$
I	Rapid	20.0	1.3	4.1	2.9	0.45:1.41:1.00
II	Rapid	40.0	3.4	8.2	6.1	0.55:1.35:1.00
III	Rapid	81.0	5.4	14.1	9.8	0.55:1.44:1.00
IV	Slow	42.0	12.0	2.8	...	4:28:1.00
V	Slow	110.0	31.2	7.1	...	4:36:1.00

$B_9H_{13}O(n$-$C_4H_9)_2$, although giving lower yields, can be carried out on large scale in the laboratory without using high-vacuum equipment.

Since decomposition of i-B_9H_{15} in the presence of a base leads exclusively to a B_9H_{13}(ligand) compound, we are led to conclude that the most probable course of the decomposition of i-B_9H_{15} is loss of a molecule of hydrogen to form the highly reactive species B_9H_{13}. This mode of decomposition is not uncommon in borane chemistry as exemplified by the well-known B_4H_{10} to B_4H_8 conversion and the proposed B_5H_{11} to B_5H_9 reaction. In the absence of the electron pair donors B_9H_{13} possibly undergoes further reaction with itself or i-B_9H_{15} to form ultimately B_8H_{12}, $B_{10}H_{14}$, and n-$B_{18}H_{22}$ (*vide infra*). In the presence of a base, B_9H_{13} is trapped without further reaction forming a B_9H_{13}(ligand) compound. The B_9H_{13} molecule has been predicted by Lipscomb to exist in an environment free of electron pair donors.[15]

The yield data in Table I suggest that B_8H_{12} and $B_{10}H_{14}$ may be initially formed in equal quantity. Although in all cases the final yield of B_8H_{12} appears to be somewhat lower than that of $B_{10}H_{14}$, it is not unreasonable to assume that some of this material was lost owing to decomposition during the isolation procedure because of the extremely unstable nature of B_8H_{12}. We suggest that B_8H_{12} and $B_{10}H_{14}$ may originate from the disproportionation of two B_9 fragments. This may be expressed by either of the equations

$$2B_9H_{13} \longrightarrow B_8H_{12} + B_{10}H_{14} \qquad (1)$$

$$B_9H_{13} + i\text{-}B_9H_{15} \longrightarrow B_8H_{12} + B_{10}H_{14} + H_2 \qquad (2)$$

While there is at the present time no way to decide between reactions 1 and 2, it would seem that since rapid decomposition favors high yields of B_8H_{12} and $B_{10}H_{14}$, the first reaction might be favored. Perhaps naively, this process may be represented as in Figure 1. The illustration shows the transfer of a boron atom from one B_9 fragment to another. The transferred boron assumes a 6,9 position in the resulting decaborane molecule.

An alternative route to the formation of B_8H_{12} and $B_{10}H_{14}$ has been suggested in the form of eq 3 and 4.

$$i\text{-}B_9H_{15} \longrightarrow B_8H_{12} + {}^1/_2B_2H_6 \qquad (3)$$

$${}^1/_2B_2H_6 + i\text{-}B_9H_{15} \longrightarrow B_{10}H_{14} + 2H_2 \qquad (4)$$

We believe these reactions to be less likely than the disproportionation mechanism because no B_2H_6 has been observed among the volatile products of the reaction and because of the quantitative conversion of i-B_9H_{15} to B_9H_{13}(ligand) compounds. Indeed it has been recently shown that reaction 3 at low temperatures appears to proceed in the direction opposite to that indicated.[16] It is interesting to note, however, that n-B_9H_{15} has recently been shown to decompose to B_8H_{12} at room temperature by a first-order mechanism.[17]

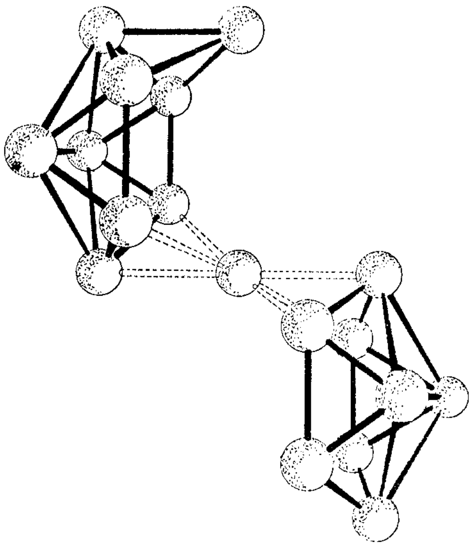

Figure 1.—Transfer of a boron atom from one B_9 fragment to another to yield B_8H_{12} and $B_{10}H_{14}$.

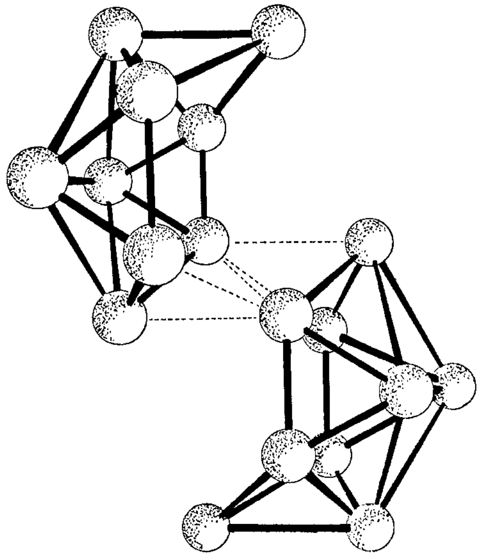

Figure 2.—Configuration of two B_9 fragments necessary to result in condensation to form n-$B_{18}H_{22}$.

The normal isomer of $B_{18}H_{22}$ appears to be the only other discrete product of the i-B_9H_{15} decomposition. We again suggest that n-$B_{18}H_{22}$ may arise through a dimerization of two B_9 fragments. This process may be represented by either eq 5 or 6.

$$2B_9H_{13} \longrightarrow n\text{-}B_{18}H_{22} + 2H_2 \qquad (5)$$

$$B_9H_{13} + i\text{-}B_9H_{15} \longrightarrow n\text{-}B_{18}H_{22} + 3H_2 \qquad (6)$$

Since slow decomposition of B_9H_{15} and consequent low concentrations of B_9H_{13} favors high yields of n-$B_{18}H_{22}$, eq 6 may be the more probable of the two. Figure 2 shows the con-

---

[15] W. N. Lipscomb, *Inorg. Chem.*, **3**, 1683 (1964).

[16] D. B. MacLean, J. D. Odom, and R. Schaeffer, *ibid.*, **7**, 408 (1968).

[17] J. F. Ditter, J. R. Spielman, and R. E. Williams, *ibid.*, **5**, 118 (1966).

figuration of two B_9 fragments necessary to result in condensation to form n-$B_{18}H_{22}$. It is important to observe that examination of a model of n-$B_{18}H_{22}$ reveals the fact that cleavage of this molecule into two B_9 fragments produces either two threefold symmetrical B_9 structures similar to i-B_9H_{15}, or two less symmetrical fragments having the boron arrangement of n-B_9H_{15}. Similarly, separation of a model of i-$B_{18}H_{22}$ into two B_9 components in all cases leads to one fragment having the threefold symmetry of i-B_9H_{15} and the other fragment having the boron structure of n-B_9H_{15}. These facts could account for the absence of i-$B_{18}H_{22}$ in the products of the decomposition of i-B_9H_{15} although it is to be noted that there are no reported physical properties for i-$B_{18}H_{22}$ and small yields might escape detection in our workshop.

It is also interesting to note that Wang, Simpson, and Lipscomb discussed formation of $B_9H_{13}L$ compounds in terms of a hypothetical B_9H_{15} molecule of C_{3v} symmetry and argued against the existence of the latter on the basis of violations of topological theory.[18] We cannot now be certain that i-B_9H_{15} has the structure suggested by them (and indeed the instability of the substance may effectively prevent more detailed structural studies than those based on nmr). If the structure is assumed to be correct, this provides an interesting measure of the strength of one assumption of topological theory: the steric effects of attaching two bridge hydrogens to a boron attached to four other nearest neighbor borons are not as severe as first considerations might suggest. If true, numerous new structures can be considered as intermediates in synthesis of boranes or in rearrangement reactions. Sufficiently delicate studies may succeed in isolating one or more additional compounds and shed additional light on an important aspect of borane chemistry.

Acknowledgments.—This work was supported at various stages by the National Science Foundation under Grant GP-4944X and the Office of Naval Research. We also thank the National Aeronautics and Space Administration for a traineeship held by P. C. K.

(18) F. E. Wang, P. G. Simpson, and W. N. Lipscomb, *J. Chem. Phys.*, **35**, 1335 (1961).

CONTRIBUTION NO. 1526 FROM THE DEPARTMENT OF CHEMISTRY,
INDIANA UNIVERSITY, BLOOMINGTON, INDIANA 47401

Studies of Boranes. XXIV. Some Chemistry of Octaborane-12 and Preparation of Octaborane-14[1]

BY JERRY DOBSON AND RILEY SCHAEFFER

Received August 11, 1967

Octaborane-12 has been found to be a strong monobasic Lewis acid; several compounds of the type $B_8H_{12}\cdot L$ were prepared. Octaborane-12 was nearly quantitatively converted to hexaborane-10 by limited hydrolysis and in about 45% yield to n-nonaborane-15 by diborane. A new octaborane, octaborane-14, was prepared from octaborane-12 in a two-step conversion.

Introduction

The earlier literature contained several reports of the probable existence of an octaborane-12 but it was not until 1964 that such a compound was isolated from the products of a discharge reaction involving diborane, pentaborane-9, and hydrogen.[2,3] This method of preparation is not satisfactory for producing workable quantities because of low yield and simultaneous production of many other boranes requiring difficult and tedious separations. Only one other compound which could be considered to be an octaborane-12 derivative has been reported, $C_2H_5NHB_8H_{11}NH_2C_2H_5$.[4-6]

In the course of recent work in this laboratory a method was developed by which one can prepare gram quantities of octaborane-12 as part of a readily separated reaction mixture. It thus seemed opportune to undertake the investigation of some of the chemistry of this little known borane.

Experimental Section

Methods.—Standard high-vacuum techniques were employed wherever possible throughout this investigation and have been treated elsewhere.[7] Direct weighing of octaborane-12 was inadvisable because of its poor thermal stability. When crude measurements of quantity were sufficient, it was convenient to measure octaborane-12 by noting the volume of frozen sample at −45° in a calibrated tube. The density reported from X-ray studies was used to calculate weight.[3] When care was taken to avoid bubbles in the sample, the accuracy of this method was found to be within about 5%. When quantitative measurements were necessary, they were obtained by difference using a tared reaction tube and accurate weights of other more easily handled reagents.

Materials.—The octaborane-12 used in this work was prepared

---

(1) For paper XXIII of this series see J. Dobson, P. C. Keller, and R. Schaeffer, *Inorg. Chem.*, **7**, 399 (1968).

(2) R. E. Enrione, P. F. Boer, and W. N. Lipscomb, *J. Am. Chem. Soc.*, **86**, 1451 (1964).

(3) R. E. Enrione, P. F. Boer, and W. N. Lipscomb, *Inorg. Chem.*, **3**, 1659 (1964).

(4) R. Lewin, P. G. Simpson, and W. N. Lipscomb, *J. Am. Chem. Soc.*, **85**, 478 (1963).

(5) R. Lewin, P. G. Simpson, and W. N. Lipscomb, *J. Chem. Phys.*, **39**, 1532 (1963).

(6) B. M. Graybill, A. R. Pitochelli, and M. F. Hawthorne, *Inorg. Chem.*, **1**, 626 (1962). This reference describes the preparation of a material thought to be $C_2H_5NH_3{}^+B_8H_{12}NH_2C_2H_5{}^-$ but which was identified as the octaborane derivative by X-ray studies.

(7) R. T. Sanderson, "High-Vacuum Manipulation of Volatile Compounds," John Wiley and Sons, Inc., New York, N. Y., 1948.