still not satisfactory; the apparent motions of the perchlorate groups are not adequately described by either model. We interpret this to mean that the perchlorate groups are moving anharmonically, and/or these ions are disordered. In support of our argument that the hydrogen bonding in this complex is generally weak, we observe that three of the eight independent perchlorate oxygen atoms have no close approaches to any of the amine hydrogen atoms and yet the apparent thermal motions of these three atoms are no greater than those of the four weakly bonded perchlorate oxygen atoms.

Registry No. $[(en)(dien)Co\text{-}O_2\text{-}Co(en)(dien)]^{4+}(ClO_4^-)_4$, 40685-52-7.

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Contribution No. 2193 from the Department of Chemistry,
Indiana University, Bloomington, Indiana 47401

Studies of Boranes. XXXVII. Some Reactions of Triborane(7) and the Structure of Triborane(7)-Carbonyl[1]

JAMES D. GLORE, JEROME W. RATHKE, and RILEY SCHAEFFER*

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The crystal and molecular structure of triborane–carbonyl, $B_3H_7 \cdot CO$, has been determined from three-dimensional X-ray data collected at $-113 \pm 10°$ by counter scan techniques. The compound crystallizes in space group $P1$ with lattice constants $a = 8.304\ (6)$ Å, $b = 5.667\ (3)$ Å, $c = 5.683\ (3)$ Å, $\alpha = 102.48\ (6)°$, $\beta = 75.96\ (7)°$, $\gamma = 104.07\ (6)°$, and a calculated density of $\rho_c = 0.907$ g cm^{-3} for $Z = 2$. All atomic coordinates were located by Patterson and Fourier techniques and full-matrix least-squares refinement of 670 observed reflections provided conventional and weighted R values of 0.0388 and 0.0432, respectively. The structure is described by the *styx* notation 1104. The physical properties of this compound are presented along with some chemistry of its reactive precursor, B_3H_7, notably the formation of B_8H_{18}.

Introduction

The reaction of dimethyl ether-triborane(7) at $-16°$ with boron trifluoride has been studied by Deever and Ritter as a source of the unstable species, B_3H_7.[2,3] Only recently have Paine and Parry used this reaction to prepare triborane(7)-carbonyl by generation of B_3H_7 in the presence of carbon monoxide.[4] Independently of these workers we have generated B_3H_7 at significantly lower temperatures by reaction of BF_3 with the more stable and more easily formed tetrahydrofuran adduct. The reaction is qualitatively similar to that reported with the dimethyl ether adduct except that B_8H_{18} is obtained in high yield. This rare hydride was not observed in the corresponding reaction of dimethyl ether-triborane(7) with BF_3. When the reaction was done in the presence of carbon monoxide, triborane(7)-carbonyl was obtained. A thorough study of the properties of this material and its crystal structure are given in this paper.

Experimental Section

Methods. Standard high-vacuum techniques as described elsewhere[5,6] were used throughout this investigation. Mass spectra were recorded on an AEI Model MS-9 mass spectrometer at 70 eV. ^{11}B and ^1H magnetic resonance spectra were recorded at $-50°$ on a Varian Associates HR 220 spectrometer at 70.6 and 220 MHz, respectively. The "line-narrowed" spectra at 70.6 MHz were obtained by means of a home-built pulse apparatus using a Nicolet 1080 computer to process the data. Infrared spectra were recorded on a Perkin-Elmer Model 621 spectrometer.

Materials. Carbon monoxide and boron trifluoride (Matheson) were purified by distillation through -196 and $-131°$ baths, respectively. Tetrahydrofuran was distilled from lithium aluminum hydride. Tetraborane(10) was prepared from tetramethylammonium triborohydride and polyphosphoric acid.[7]

Preparations. Tetraborane(10) (6.94 mmol) and tetrahydrofuran (28 mmol) were allowed to react in a heavy-walled glass bomb tube of 100-ml volume to form $THF \cdot B_3H_7$ similar to a method described elsewhere.[8] After pumping off excess tetrahydrofuran and B_2H_6 at 0°, BF_3 (13.1 mmol) and CO (35 mmol) were condensed in at $-196°$. The bomb tube was sealed and completely submerged in a $-45°$ bath for 1.5 hr during which time the solid $THF \cdot B_3H_7$ liquefied. Excess CO was removed at $-196°$ and volatile materials were pumped from the tube at $-45°$ for 2 hr and at 0° for 1 additional hr. Infrared spectroscopic examination of the colorless liquid remaining in the bomb, $BF_3 \cdot THF$ (6.45 mmol), showed that it contained no boron hydrides. Triborane–carbonyl (2.14 mmol) passed a $-78°$ bath and formed crystals in a $-112°$ bath. This material was contaminated with B_4H_{10} and B_5H_{11} as judged from the ^{11}B nmr spectrum. Attempts to purify the material further by trap to trap distillation failed because of extensive decomposition during transfer. However, low-temperature column[9] sublimination at $-100°$ gave samples of high purity; typical yields after column fractionation varied from 28 to 30% based on starting B_4H_{10}. Also obtained in the reaction were BF_3 (6.84 mmol), B_4H_{10} (1.30 mmol), B_5H_{11} (0.18 mmol), B_6H_{12} (0.14 mmol), and B_8H_{18} (0.42 mmol). No other carbonyl-boranes and only a trace of B_2H_6 were observed.

When B_3H_7 was generated as above but without addition of CO, 0.378 g of B_4H_{10} gave 0.112 g of B_8H_{18}. Also observed were B_6H_{12}, B_5H_{11}, and B_4H_{10} as well as much smaller amounts of B_5H_9 and B_2H_6. Only 0.029 mmol of H_2 was isolated from the reaction. When the reaction was carried out at 0° with a reaction time of 15 min, 1.14 g of B_4H_{10} resulted in 0.261 g of B_8H_{18}. The lower yield at this temperature is accounted for by decomposition as evidenced by the yellow coloration of the reaction mixture. The boron hydrides isolated in these reactions were identified by their characteristic ir and ^{11}B nmr spectra.

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Table I

	x[a]	y	z	B_{11} (or B)[b]	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
O_1	0.169 (0)	0.800 (0)	0.005 (0)	2.0 (1)	1.6 (2)	1.0 (1)	0.6 (2)	−1.0 (1)	−0.7 (2)
C_2	0.753 (1)	0.046 (1)	0.888 (1)	0.8 (1)	0.5 (1)	1.9 (2)	−0.2 (2)	0.2 (2)	−0.1 (2)
B_1	0.355 (1)	0.171 (1)	0.257 (2)	0.8 (1)	1.3 (2)	3.2 (3)	0.9 (3)	−1.5 (3)	−0.3 (4)
B_2	0.224 (1)	0.418 (2)	0.377 (2)	1.4 (1)	1.3 (2)	2.6 (3)	1.2 (3)	0.7 (3)	1.1 (4)
B_3	0.240 (1)	0.228 (1)	0.576 (1)	0.7 (1)	2.3 (2)	2.2 (2)	1.0 (2)	−0.5 (2)	−2.4 (4)
O_2	0.831 (0)	0.201 (0)	−0.005 (0)	0.9 (0)	3.6 (2)	4.0 (2)	0.3 (2)	−1.5 (2)	−0.1 (3)
C_1	0.249 (1)	0.953 (2)	0.111 (1)	1.3 (1)	4.7 (3)	1.5 (2)	3.2 (3)	−0.5 (3)	1.7 (4)
B_4	0.642 (1)	0.827 (2)	0.740 (1)	1.0 (1)	2.6 (3)	0.7 (2)	0.3 (3)	0.0 (2)	0.3 (4)
B_5	0.764 (1)	0.591 (2)	0.620 (1)	1.0 (1)	2.5 (2)	2.5 (3)	1.3 (3)	−2.2 (3)	−0.5 (4)
B_6	0.765 (1)	0.782 (2)	0.420 (1)	1.9 (2)	2.8 (3)	1.2 (2)	0.5 (3)	−0.5 (3)	2.5 (4)
H_1	0.582 (7)	0.690 (11)	0.801 (11)	1.32 (1.25)					
H_2	0.556 (7)	0.927 (9)	0.698 (9)	0.0 (0.94)					
H_3	0.860 (8)	0.624 (11)	0.691 (11)	3.35 (1.34)					
H_4	0.667 (10)	0.395 (16)	0.629 (13)	3.39 (1.95)					
H_5	0.855 (11)	0.645 (13)	0.406 (15)	2.22 (1.89)					
H_6	0.841 (7)	0.960 (10)	0.364 (10)	2.31 (1.19)					
H_7	0.671 (7)	0.725 (10)	0.312 (10)	0.0 (1.1)					
H_8	0.447 (6)	0.273 (9)	0.113 (9)	0.5 (0.98)					
H_9	0.418 (9)	0.092 (12)	0.361 (12)	3.87 (1.57)					
H_{10}	0.132 (7)	0.360 (10)	0.248 (11)	0.0 (1.1)					
H_{11}	0.302 (7)	0.606 (10)	0.413 (9)	0.0 (0.93)					
H_{12}	0.156 (8)	0.156 (10)	0.596 (9)	0.0 (1.0)					
H_{13}	0.130 (6)	0.049 (9)	0.591 (9)	0.0 (0.91)					
H_{14}	0.323 (8)	0.333 (12)	0.722 (11)	4.07 (1.56)					

[a] Uncertainties in the final digit (standard deviations) are given in parentheses. [b] Anisotropic values are $\times 10^2$; isotropic B's are given with the uncertainty of the value in parentheses.

Vapor Pressures and Melting Points. A small sample of pure $B_3H_7\cdot CO$ in a sealed tube attached to a 6-in. U-bend manometer was observed to evolve noncondensable gas too rapidly for accurate vapor pressure measurements even at −63°. It was found that decomposition occurred on the warm parts of the manometer at this temperature since noncondensable gas was not evolved when the entire apparatus was submerged in the −63° bath. Similarly, the sample did not evolve noncondensable gas when completely submerged in a −45° bath but did so at −30°. Since mercury freezes at −39°, vapor pressure measurements over an appreciable temperature range were impossible with a conventional mercury system. For this reason vapor pressure measurements were taken using a glass Bourdon gauge as a null-pressure indicator.[6] All parts of the gauge exposed to sample were submerged in a low-temperature bath whose temperature was determined with an NH_3 vapor pressure manometer. The pressure of air which balanced the Bourdon gauge was measured to 0.1 mm with a cathetometer. The sensitivity of the gauge was approximately 1 mm deflection/1.6 mm. The following vapor pressures were recorded in mm: 7.8 (−49.0°), 10.7 (−45.3°), 15.5 (−40.5°), 21.6 (−36.7°). A least-squares fit to the data resulted in the equation $\ln P = -4349/T + 21.45$. Vapor pressures calculated from this equation at these temperatures, respectively, are 7.8, 10.7, 15.8, 21.3 mm. The calculated boiling point is 20.3°, the heat of vaporization 8640 cal/mol, and the Trouton constant 29 cal/deg mol.

Stock melting points of highly purified samples were −93.0, −93.3, −93.0, and −93.7°.

Spectroscopic Data. The infrared spectrum of $B_3H_7\cdot CO$ was measured near liquid nitrogen temperature in a low-temperature cell equipped with an Irtran cold window. The following bands were recorded in cm^{-1} in the region of useful transmission of the cell (4000–700 cm^{-1}): 2531 (s), 2460 (s), 2325 (w), 1982 (w), 1881 (w), 1658 (sh), 1635 (m), 1174 (m), 1158 (s), 1023 (m), 986 (w), 927 (sh), 917 (m), 883 (w), 791 (w), 776 (w). A very strong carbonyl stretching band was observed at 2203 cm^{-1}.

The m/e values (relative intensities) of the major peaks in the mass spectrum follow: 66 (11), 65 (9), 39 (7), 38 (42), 37 (36), 36 (26), 35 (30), 34 (16), 33 (8), 28 (100), 27 (51), 26 (−15), 25 (22), 24 (44), 23 (22). The highest mass peak had an exact mass of 66.0613; that calculated for $^{11}B_3H_5{}^{12}C^{16}O$ was 66.0619. In most cases peaks with intensities less than 10% of base peak are not given because of extensive decomposition in the inlet system. This occurred even though the sample was evaporated from a −80° bath through a direct inlet to the source of the spectrometer.

The 70.6-MHz ^{11}B nmr spectrum of a neat sample at −50° consists of a low-field triplet of area 2 (δ +6.39 ppm ($BF_3\cdot Et_2O$), $J_{BH} = 127$ Hz) and a high-field triplet of area 1 (δ +52.6 ppm, $J_{BH} = 100$ Hz) in agreement with the previously reported 32.1-MHz spectrum.[4] Artificial "line narrowing" of the ^{11}B spectrum resolves the bridge proton–boron coupling of 43 Hz. The "line-narrowing" technique applied to ^{11}B nmr spectroscopy is described elsewhere.[10] The 220-MHz ^1H nmr spectrum in chloroform solution consists of two partially overlapping quartets and a broad singlet with respective chemical shifts of −2.47 ($J = 122$ Hz, area 4), −1.40 ($J = 98$ Hz, area 2), and +1.76 (area 1) in ppm relative to internal TMS.

X-Ray Data Collection. A sample of purified $B_3H_7\cdot CO$ was condensed into a thin-walled Pyrex capillary (o.d. ~0.5 mm), sealed *in vacuo* and mounted on a Picker FACS-1 diffractometer which was fitted with a gaseous N_2 sample-cooling system.[11] At no time during sample handling did the temperature in the capillary reach the melting point of $B_3H_7\cdot CO$ (−93°). A single crystal of triclinic habit was grown *in situ* and the temperature was then lowered an estimated 20°. This crystal had approximate dimensions 0.35 × 0.35 × 0.50 mm and ω scans of four intense reflections showed no anomalies and gave an average half-width of 0.16°. The hemisphere bounded by $\rho_{max} = 0.024$ was systemically searched and all reflections located could be indexed in a triclinic system. A least-squares fit to 12 carefully centered high-order reflections gave the following cell parameters and standard deviations: $a = 8.304$ (6), $b = 5.667$ (3), $c = 5.683$ (3) Å; $\alpha = 102.48$ (6), $\beta = 75.96$ (7), $\gamma = 104.07$ (6)°. These parameters are also consistent, within experimental error, with a c-centered monoclinic cell having $a = 8.851$, $b = 7.106$, $c = 8.304$ Å, and $\beta = 108.15°$; however monoclinic symmetry was ruled out when the data indexed in this system failed to show the required equivalence of intensities for the hkl and $\bar{h}k\bar{l}$ or hkl and $h\bar{k}l$ pair. Thus the space group is either $P1$ or $P\bar{1}$. The calculated density for two molecules per unit cell ($V = 248.12$ Å^3) is $\rho_{calcd} = 0.907$.

Data were collected by automation using Mo Kα radiation with a highly oriented graphite monochromater and a takeoff angle of 2.1°. Reflections were scanned at 2° min^{-1} over a 2° range symmetrically disposed about the calculated peak center, and a 10-sec stationary background count was taken at both ends of the scan. Intensities of three standard reflections were checked every 50 readings. The standards showed a uniform decrease in intensity over a 6–8-hr period and the crystal was realigned when the decrease reached 10%. When realignment could no longer restore the intensity of the standards to at least 90% of their original values, the crystal was melted and regrown. The new crystal was very nearly identical with the original in size and orientation, and after thermal equilibrium was reached, the intensities of the standard reflections were found to have been restored. In all, three crystallizations and several realignments were required to collect data to 55° on 2θ. A full sphere of data bounded by $\rho_{max} = 0.432$ was collected and collection of a redundant hemisphere was nearly completed when the capillary fractured and the sample decomposed.

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The 3806 reflections collected were corrected for background and polarization but not extinction or absorption ($\mu = 0.59$ cm^{-1}, maximum correction 0.999). Due to the unexpected loss of the crystal, very strong reflections could not be recollected using attenuators. However, examination of a strip chart recording of the rate meter taken during the data collection showed that serious counter overflow (>20,000 cps) was significant for at most 13 reflections. These strong reflections were included in the data as if they were accurate. The data were divided into sets bounded by recrystallizations or realignments. Each set was then corrected for decrease in intensity by obtaining a least-squares plot through the standard intensities of that set and applying the inverse slope of this plot to the data set. Last, each corrected set was scaled to the first set by applying appropriate scale factors determined from the average intensities of the standards. Equivalent reflections were averaged to give 1149 unique reflections. The numbers of reflections whose standard errors were greater than 2.0 and 1.0 times the average standard deviations of intensities of equivalent reflections were 6 and 349, respectively. An attempt was made at this point to differentiate between the centric and acentric space groups. A zero-moment test of the data coincided with the theoretical acentric curve for values near the average intensity but diverged to the centric curve for other values. The distribution of the normalized structure factors favored the centric case for E's > 2. In view of the ambiguity of the space group selection, the data were refined in both groups, starting with $P\bar{1}$.[12]

Solution of the Structure. A sharpened Patterson map was obtained from which a B–C–O unit was located. These three atomic positions were used in a least-squares refinement which minimized $\Sigma w(F_o - F_c)^2$ where $w = 1/(F_o + 0.02F_o^2)$ and F_o and F_c are the observed and calculated structure factors, respectively. Scattering factors used were those of Cromer and Waber.[13] The two remaining heavy atoms were located from an F_o Fourier synthesis, and a difference Fourier map located all seven hydrogen atoms. Four cycles of refinement using anisotropic temperature factors for the five heavy atoms and isotropic temperature factors fixed at 2.0 (the approximate average isotropic value of the heavy atoms) resulted in conventional and weighted R values of 0.1192 and 0.2169, respectively. Restriction of the data of those 670 reflections for which $F_o^2 \geqslant 2.33\sigma$ (where σ is the greater of the average σ for equivalent reflections or the standard deviation of the average intensity for equivalent reflections) caused convergence to R values of 0.0473 and 0.0576 after three cycles of refinement.

The data were refined in space group $P1$ using the atomic positions of the $P\bar{1}$ refinement and their symmetry equivalents, the origin being fixed by holding the position of the first oxygen constant. After five cycles of refinement the R values converged to 0.0388 and 0.0432. The largest positional shifts during the final cycle were for the x coordinates of B_5 and H_{10}, 0.62σ and 0.72σ, respectively (0.005 and 0.042 Å). A difference Fourier map showed no peak larger than 0.25 times the smallest hydrogen peak (*i.e.*, not larger than approximately 0.2 e/Å^3). The hydrogen B values were allowed to vary for three cycles but this refinement failed significantly to reduce the R values, nor did any of the B values exceed 4.1. The final positional and thermal parameters for $B_3H_7 \cdot CO$ are listed in Table I. Table II lists the final values for $10F_o$ and $10F_c$ for the 670 observed reflections.[14]

Results and Discussion

The major products formed when $B_3H_7 \cdot$THF is treated with excess BF_3 at −45° are $BF_3 \cdot$THF, B_8H_{18}, B_6H_{12}, B_5H_{11}, and B_4H_{10}. Lesser amounts of B_5H_9 and B_2H_6 are also produced. Isolation of B_8H_{18} in high yields at −45° and somewhat lower amounts at 0° coupled with the failure to observe this product in the reaction of the dimethyl ether adduct of triborane with BF_3 at −16°[2] suggests that the course of the reaction is ligand dependent. Furthermore, the role of B_2H_4 in the reactions of B_3H_7 may be more important than originally suspected, particularly in view of the recent observations of the facile cleavage of adducts of triborane(7) with weakly basic ligands to form diborane(4) adducts in the presence of dimethyl ether.[4,15,16] Indeed, $B_3H_7 \cdot CO$ decomposes at 0° to form $B_2H_4 \cdot 2CO$ without added ligand.[17]

Table III

	Molecule I	Molecule II
Distances, Å		
C–O	1.11 (1)	1.12 (1)
C–B_1	1.54 (1)	1.57 (1)
B_1–B_2	1.89 (1)	1.85 (1)
B_1–B_3	1.83 (1)	1.80 (1)
B_2–B_3	1.77 (1)	1.73 (1)
Angles, deg		
O–C–B	177.9 (7)	179.0 (6)
C–B_1–B_2	107.3 (5)	106.1 (5)
C–B_1–B_3	109.5 (6)	106.6 (5)
B_2–B_1–B_3	56.6 (4)	56.6 (4)
B_1–B_2–B_3	59.9 (4)	60.0 (5)
B_1–B_3–B_2	63.5 (5)	63.4 (5)
Distances, Å		
B_1–H_1	1.09 (8)	1.10 (6)
B_1–H_2	1.13 (5)	1.12 (5)
B_2–H_3	1.13 (5)	0.93 (8)
B_2–H_4	1.18 (5)	1.17 (8)
B_2–H_5	1.30 (5)	1.35 (7)
B_3–H_5	1.22 (5)	1.19 (7)
B_3–H_6	1.16 (7)	1.13 (7)
B_3–H_7	1.19 (5)	1.05 (5)
Angles, deg		
H_1–B_1–H_2	112 (5)	115 (4)
H_3–B_2–H_4	124 (4)	104 (4)
H_6–B_3–H_7	130 (5)	119 (7)
B_1–H_1–B_3	69 (4)	67 (3)
B_2–H_5–B_3	89 (3)	85 (5)

Reaction of $B_3H_7 \cdot$THF with excess BF_3 and CO at −45° yields $BF_3 \cdot$THF, $B_3H_7 \cdot CO$, B_8H_{18}, B_4H_{10}, B_6H_{12}, B_5H_{11}, and a trace of B_2H_6. Isolation of $B_3H_7 \cdot CO$ in high yields lends strong support to the assumption that free B_3H_7 is produced as a first step in the reaction. Other reaction products are reminiscent of those formed in the reaction with no CO and may be indicative of competitive pathways in the presence of CO. The spectroscopic data for triborane(7)-carbonyl are presented in the Experimental Section and are in excellent agreement with the 1104 triborohydride(8)-like structure proposed by Paine and Parry.[4] This structure is confirmed by the crystallographic study.

Triborane-carbonyl is assigned to the space group $P1$ based on Hamilton's test.[18] Refinement of 670 reflections converged to an R of 0.0473 for 74 parameters ($P\bar{1}$) and to 0.0383 for 147 parameters ($P1$). Using Hamilton's method, one calculates $\mathcal{R}_{73,532,0.005} = 1.097$. Because the observed R factor ratio of 1.219 exceeds this value, the hypothesis that the true space group is $P\bar{1}$ can be rejected at a confidence level greater than 99.5%. Assignment to $P1$ is not a function of the weighting scheme. Refinement using unit weight and σ of F_o as weights gave R factor ratios exceeding 1.097 in both cases. The deviation from the centric case, while statistically significant, is actually very small. The apparent center is at 0.5000, 0.5066, 0.4994 for five heavy-atom pairs and at 0.4973, 0.5042, 0.4999 for all atom pairs. The near

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(12) Preliminary data were processed using locally written programs. Least-squares, Fourier, and subsequent calculations were performed using programs written by Dr. A. Larson of Los Alamos Scientific Laboratory, Los Alamos, N. M.

(13) D. T. Cromer and J. T. Waber, *Acta Crystallogr.*, 18, 104 (1968).

(14) Table II, a listing of observed and calculated structure factors, will appear following these pages in the microfilm edition of this volume of the journal. Single copies may be obtained from the Business Operations Office, Books and Journals Division, American Chemical Society, 1155 Sixteenth St., N.W., Washington, D. C. 20036. Remit check or money order for $3.00 for photocopy or $2.00 for microfiche, referring to code number INORG-73-2175.

(15) W. R. Deever and D. M. Ritter, *J. Amer. Chem. Soc.*, 89, 5073 (1967).

(16) E. R. Lory and D. M. Ritter, *Inorg. Chem.*, 10, 939 (1971).

(17) J. Rathke and R. Schaeffer, submitted for publication.

(18) W. C. Hamilton, *Acta Crystallogr.*, 18, 502 (1965).

approach to a center of inversion, together with the decrease from 9.1 to 4.6 observations per variable on going from $P\bar{1}$ to $P1$, makes us uncomfortable with the latter space group, despite the high statistical significance which militates its selection.

A perspective drawing of the molecule is shown in Figure 1; the principal intramolecular distances and angles are given in Table III. The only significant nonbonded interactions are between H_1 and B_3. These distances are 1.90 (6) and 1.90 (8) Å in the first and second molecules, respectively. The three boron atoms in each molecule define a plane which nominally bisects the terminal BH_2 angles. The bridge hydrogens are displaced 0.064 and 0.234 Å from these planes in the first and second molecules, respectively. The least-squares plane passed through O-C-B and bridge hydrogen makes a dihedral angle of 93.2° with the plane of the three boron atoms in the first molecule and the corresponding value is 91.1° in the second molecule. Thus the molecule closely approaches C_s symmetry in the solid state.

From the data in Table III it is clear that neither of the hydrogens attached to the unique boron atom is within bridging distance of the remaining boron atoms and therefore the structure is unambiguously described by the *styx* notation 1104 if the CO group is replaced with a H^- ion. Previous structure determinations for B_3 molecules give the 2013 structure for $B_3H_8^-$ [19] and a structure intermediate between 2013 and 1104 for $B_3H_7N(CH_3)_3$.[20] It is quite possible that the preference for a 2013 structure is a function of the strength of σ donation of the ligand. Structural parameters for determinations of B_3 molecules are summarized in Table IV.

Summary

Reaction of $B_3H_7\cdot THF$ with BF_3 at −45° provides an excellent synthetic route to B_8H_{18}. In the presence of CO, $B_3H_7\cdot CO$ is formed in very good yields. This latter compound has the 1104 structure as confirmed by an X-ray crystallographic study.

Registry No. $B_3H_7\cdot CO$, 40685-59-4; $B_3H_7\cdot THF$, 12544-89-7; BF_3, 7637-07-2; CO, 630-08-0; B_8H_{18}, 12421-66-8; boron-11, 14798-13-1.

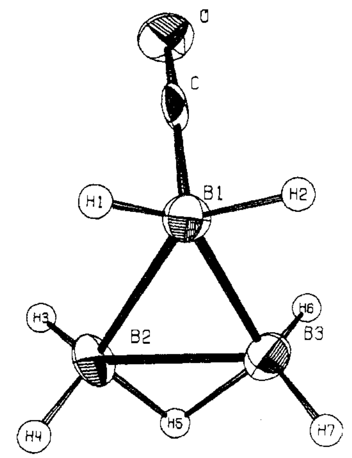

Figure 1. Perspective drawing of triborane(7)-carbonyl, prepared by ORTEP (C. K. Johnson, Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1965).

Table IV

	$B_3H_8^-$ [a]	$B_3H_7\cdot N(CH_3)_3$ [b]	$B_3H_7\cdot CO$	
		Distances, Å		
B-B	1.766	1.744	1.767	1.732
	1.767	1.820	1.832	1.797
	1.797	1.803	1.895	1.854
B-H	1.037	1.09	1.091	0.927
(terminal)	1.143	1.11	1.106	1.046
	1.156	1.12	1.118	1.130
	1.157	1.12	1.127	1.134
	1.157	1.14	1.157	1.168
	1.184	1.18	1.178	1.189
B-H	1.554, 1.184	1.39, 1.23	1.354, 1.194	
(bridge)	1.554, 1.184	(1.75, 1.12)	1.301, 1.219	
		Angles, Deg		
B-H-B	79.1	83.27	85.40	
	79.1		89.02	
B-B-B	61.1	61.71	63.50	63.37
	59.7	60.74	59.92	60.01
	59.4	57.56	56.58	56.62

[a] W. N. Lipscomb, "Boron Hydrides," W. A. Benjamin, New York, N. Y., 1963, p 232. [b] Reference 19.

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