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The Crystal Structure of Cesium Tetramethylammonium Tridecahydroundecaborate

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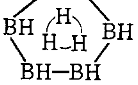
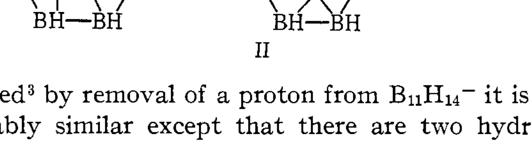
The crystal structure of cesium tetramethylammonium tridecahydroundecaborate, $CsN(CH_3)_4B_{11}H_{13}$, has been determined using counter-recorded Mo $K\alpha$ diffraction data. The structure is orthorhombic, with space group Ccmb and a unit cell having $a = 11.44$ A, $b = 11.58$ A, $c = 24.78$ A, and $Z = 8$. The final R is 5.8%. The $B_{11}H_{13}^{2-}$ ion has mirror symmetry in the crystal and contains a boron framework consisting of a nearly regular icosahedron missing one vertex. Two of the hydrogen atoms bridge nonadjacent edges of the open pentagonal face; the remainder are apical. The hydrogen-bridged B–B bond length is 1.89 ± 0.013 A, the lengths of the other B–B bonds on the open face are 1.80 ± 0.013 and 1.82 ± 0.013 A, and the remaining B–B bonds range from 1.72 to 1.83 A in length.

Introduction

The similar ions $B_{11}H_{14}^-$ and $B_{11}H_{13}^{2-}$ are apparently subtly different examples of the relatively rare case among boron hydrides where the nuclear magnetic resonance spectra are simpler than would be expected for any imaginable static structure. Aftandilian, *et al.*,[3] who first prepared $B_{11}H_{14}^-$ and $B_{11}H_{13}^{2-}$, examined the H^1 and B^{11} nmr spectra of the former and found basic agreement with structure I which had been proposed by Moore, Lohr, and Lipscomb.[4] In structure I the BH pentagon represents the open face of a $B_{11}H_{11}$ cage having icosahedral symmetry except for the missing vertex at the open face, and the three additional hydrogen atoms are supposed by Aftandilian, *et al.*, to attain equivalence in their nuclear resonance energies by nearly free rotation about the fivefold axis. If only the conventional bridge and terminal bonding structures for hydrogen are considered and if the argument of Moore, *et al.*, is accepted that bonding on the open face will involve three bonding molecular orbitals formed from five boron orbitals pointed inward and upward and the orbitals provided by the hydrogen atoms, one must suppose I to be an average of structures of type II. Because the $B_{11}H_{13}^{2-}$ ion is reversibly formed[3] by removal of a proton from $B_{11}H_{14}^-$ it is presumably similar except that there are two hydrogen atoms over the open face.



I



II

If the instantaneous structures II are correct, then the two B_{11} ions appear to be unusual examples of intramolecular exchange in that one but only one of the two hydrogen atoms of each instantaneous BH_2 group participates in the exchange. In previous cases where internal exchange or tautomerism has been proposed, either all protons become equivalent through exchange ($Al(BH_4)_3$[5] and $B_3H_8^-$)[6] or only bridge protons move (B_6H_{10},[7–9] Figure 1).

The formal method[4] of constructing $B_{11}H_{14}^-$ from $B_{12}H_{12}^{2-}$ by replacement of apical BH with triangular H_3^+ suggests that direct H···H interaction might play a role in lowering the barrier to tautomerism and might simultaneously explain the participation in exchange of only one of the protons in the formal BH_2 group. To verify a structure of type II for $B_{11}H_{14}^-$ and an analogous form for $B_{11}H_{13}^{2-}$, and to investigate the possibility of even weak H···H interaction, the crystal structure of $CsN(CH_3)_4B_{11}H_{13}$ has been determined.

Experimental Section

A sample of the blocklike crystals was supplied by Dr. F. Klanberg of Du Pont. Most of the crystals had a frosted appearance, but the surface could be cleaned by rolling them between the fingers. The cleaned crystals were almost perfectly transparent. Approximately cubic specimens about 0.2–0.4 mm on a side were used to make early photographs. These precession and Weissenberg photographs, prepared respectively with Mo $K\alpha$ and Cu $K\alpha$ radiation, first seemed to indicate orthorhombic symmetry, a unit cell about $5.7 \times 5.8 \times 25$ A, and absences $0kl$ with l odd. After collection of intensity data, it was discovered that a and b were in fact double the values originally measured. The true values, representing averages of measurements made on a Picker diffractometer and on Mo $K\alpha$ precession photographs, are $a = 11.44 \pm 0.01$ A, $b = 11.58 \pm 0.01$ A, and $c = 24.78 \pm 0.02$ A. If eight molecules are assumed to occupy one unit cell, the density is 1.36 g/cm³. The general absences, with the correct unit cell, are hkl with $h + k$ odd; $0kl$, with either k or l odd; and $hk0$, with h or k odd. These absences imply a C-centered cell, with a c or n glide perpendicular to **a** and an a or b glide perpendicular to **c**. Reference to the literature[10a] shows that two

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[1] Contribution No. 1290 from Central Research Department, E. I. du Pont de Nemours and Co.

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[3] V. D. Aftandilian, H. C. Miller, G. W. Parshall, and E. L. Muetterties, *Inorg. Chem.*, **1**, 734 (1962).

[4] E. B. Moore, Jr., L. L. Lohr, Jr., and W. N. Lipscomb, *J. Chem. Phys.*, **35**, 1329 (1961).

[5] R. A. Ogg and J. D. Ray, *Discussions Faraday Soc.*, **19**, 239 (1955).

[6] W. N. Lipscomb, *Advan. Inorg. Chem. Radiochem.*, **1**, 117 (1959).

[7] R. E. Williams, *J. Inorg. Nucl. Chem.*, **20**, 198 (1961).

[8] R. E. Williams, S. G. Gibbins, and I. Shapiro, *J. Chem. Phys.*, **30**, 333 (1959).

[9] F. L. Hirshfeld, K. Eriks, R. E. Dickerson, E. L. Lippert, Jr., and W. N. Lipscomb, *ibid.*, **28**, 56 (1958).

[10] "International Tables for X-ray Crystallography," International Union of Crystallography, Kynoch Press, Birmingham, England, 1952: (a) Vol. I; (b) Vol. III.

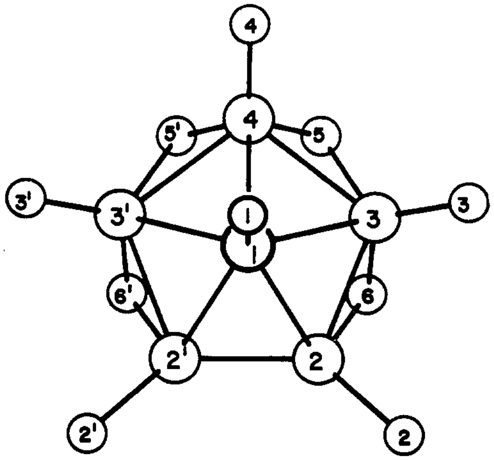

Figure 1.—Structure of B_6H_{10}, from ref 9.

space groups, one in either of two orientations, fit these requirements. These are Ccmb (No. 64) and Ccca or Cccb (No. 68). The second has eightfold general positions only of twofold (C_2) or $\bar{1}$ (C_i) symmetry, whereas the first also has such positions with symmetry m (C_s). The $B_{11}H_{13}^{2-}$ ion is very unlikely to have C_2 or C_i symmetry; hence Ccmb is the most likely space group. The first Fourier map, phased by the cesium atoms and based on the pseudo-structure obtained by ignoring the odd *h*, odd *k* reflections, led to the same conclusion. That Ccmb is the proper space group has been verified by the final refinement. The nonstandard orientation was used for convenience in relating the true and pseudo structures. The origin is the same as that in ref 10, the intersection of the mirror plane and twofold axis.

The intensity data were collected with two crystals and two different Picker card-controlled diffractometers, but using identical procedures. Mo Kα radiation was used in both cases. The data with *h* (and *k*) even were collected at Du Pont using a nearly cubic crystal 0.2 mm on a side. A scintillation detector was used to make the measurements, in conjunction with a pulse-height analyzer set to receive about 90% of the Kα energy. A zirconium filter was employed. The scanning technique was the θ–2θ scan of Furnas,[11] with a scan range of 2° and a rate of 1°/min. A 20-sec background count was made at either end of the scan range. Two standard reflections were measured about every 4 hr. Scanning covered all *h*-even reflections with $\sin\theta/\lambda \leqslant 0.7$ ($2\theta \leqslant 59.6°$) and yielded 1113 observed and one unobserved reflections. Unobserved reflections are those with $I_{\text{measd}} < 2\sigma_I$, where σ_I is derived from integrated intensity, background, and scale uncertainties by application of the usual propagation-of-error equation. The errors in background and integrated intensities were assumed to be the square roots of the respective numbers of counts, and the error in scale to be 4%. The one unobserved reflection with *h* even and those with *h* odd were assigned threshold values of $2\sigma_I$ and were included in refinement and *R*-value calculations only if $|F_c|$ exceeded $|F_{\text{th}}|$.

The reflections having *h* (and *k*) odd were collected later at Tulane when it was discovered that the unit cell was actually twice as large in the *a* and *b* directions as originally supposed. These data were measured first on a rather large crystal by the visual multiple-film equiinclination Weissenberg technique, and then on a crystal about 0.15 × 0.2 × 0.3 mm in size by the counter technique described above, except that three standard reflections were used to determine the scale factor. The crystal used for estimation of data in the Weissenberg technique was 0.6 mm long, mounted on the *a* axis. The *nkl* photographs with *n* = 1, 3, and 5 were prepared with Cu Kα radiation. These data were used in refinement for several cycles but were disregarded when the more accurate set of counter data became available.

No absorption corrections were applied to either set of data. The structure factors, $|F_o|$, were derived by application of the usual Lorentz and polarization factors. Their individual standard deviations, derived from σ_I, were used in weighting, with $w = 1/\sigma_F^2$.

Determination and Refinement of the Structure

Because of the early overlooking of the weak reflections having odd *h* and odd *k*, the structure was solved in two phases. The pseudo-structure obtained by halving *a* and *b* has orthorhombic symmetry with Pcmm, Pc2m, and Pcm2 the most probable space groups. There are two molecules per unit cell. The one heavy Cs–Cs vector in the Patterson map at (0.33, 0, $^1/_2$) showed no elongation suggestive of composite nature, and hence indicated that heavy-atom phasing of a Fourier synthesis could not give a map with other than Pcmm symmetry. This map was calculated and after study was found to contain seven peaks representing the independent half of a B_{11} cage lying across a mirror of symmetry. No indication of the tetramethylammonium ion was found. The *R* factor was 18.0% with only cesium in the structure.

The space group considered most probable for the true structure, Ccmb, requires the space group of the pseudo-structure to be Pcmm. The seven boron atoms were thus added to a model of this symmetry and a difference map was calculated. The *R* value for this calculation was 12.4%. The difference map showed four large peaks, one heavier than a boron atom, one the same size, and two about three-quarters this size. These atoms, two of which in the pseudo-structure nearly coincided with boron peaks, represented the nitrogen and three independent carbon atoms of the tetramethylammonium group. The low electron density associated with the carbon atoms is consistent with their great thermal motion. The *R* value with all atoms (except hydrogen) present was 10.1%. Three least-squares cycles in which anisotropic thermal parameters for the cesium atom and isotropic thermal parameters for the lighter atoms, as well as positional parameters of the nonsuperimposed atoms, were refined led to an *R* value of 5.6%. A difference map computed at this stage showed positive areas in the regions expected for terminal hydrogen atoms in the boron cage, but because of extensive overlap with the tetramethylammonium group in the pseudo-structure, these atoms were not included in structure-factor calculations at this time.

The *h*1*l*, *h*3*l*, and *h*5*l* photographic data had by now been measured. Addition of these data to an ordered structure having Ccmb symmetry gave an *R* of 6.1%. Three refinement cycles reduced *R* to 5.8%. Although the *R* factor for the data with odd *h* was about 20%, the pattern of agreement was good enough to prove the correctness of the basic structure. The high *R* factor at this point was due to the difficulty of measuring these weak reflections and to the fact that they are due almost entirely to scattering by the lighter atoms, several of which had had to be fixed in position. Further refinement was not attempted until a single set of data measured by counter was available.

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[11] T. Furnas, "Single Crystal Orienter Instruction Manual," General Electric Co., Milwaukee Wisc., 1956.

TABLE I

FINAL ATOMIC PARAMETERS

Atom	x (σ_x)[a]	y (σ_y)[a]	z (σ_z)[a]	B (σ_B)[a]
Cs^+(1)	0.08271 (5)	1/4	1/4	*b*
B(2)	0.0411 (12)	0	0.6796 (5)	2.6 (2)
B(3)	0.1680 (8)	0.0762 (8)	0.6990 (4)	2.5 (1)
B(4)	0.2962 (14)	0	0.6734 (6)	2.8 (2)
B(5)	0.0847 (7)	0.1258 (8)	0.6423 (4)	2.7 (1)
B(6)	0.2421 (8)	0.1266 (8)	0.6391 (4)	2.9 (1)
B(7)	0.1679 (8)	0.0784 (9)	0.5802 (4)	3.1 (2)
B(8)	0.2858 (12)	0	0.6026 (6)	2.8 (2)
N(9)	0.3176 (9)	0	0.4159 (5)	3.2 (2)
C(10)	0.3575 (18)	0	0.3576 (8)	5.9 (4)
C(11)	0.1897 (21)	0	0.4229 (10)	7.6 (5)
C(12)	0.3732 (12)	0.1054 (11)	0.4438 (5)	5.0 (2)
H(13)	−0.030 (10)	0	0.704 (5)	*c*
H(14)	0.175 (10)	0.120 (10)	0.745 (5)	
H(15)	0.383 (10)	0	0.696 (5)	
H(16)	0.025 (10)	0.213 (10)	0.644 (5)	
H(17)	0.288 (10)	0.213 (10)	0.642 (5)	
H(18)	0.173 (10)	0.125 (10)	0.543 (5)	
H(19)	0.373 (10)	0	0.579 (5)	
H(20)	0.065 (10)	0.075 (10)	0.596 (5)	

[a] Figures in parentheses are the standard deviations applicable to the final digit tabulated. No standard deviation is given for parameters fixed by symmetry. For hydrogen atoms the error is a subjective estimate based on the appearance of the difference map in which they were found and past experience; for the remaining atoms, the standard deviations are from standard treatment of the least-squares residual. [b] The temperature factor for Cs^+(1) is $\exp[-0.00556(4)h^2 + 0.00494(3)k^2 + 0.001278(8)l^2 + 0.00009(8)kl]$. [c] B is 0.8 for all hydrogen atoms.

Six cycles of refinement with the counter data produced convergence of the structure at 6.1%, with only the parameters refined earlier being allowed to vary. A difference map revealed all the hydrogen atoms in the $B_{11}H_{13}^{2-}$ ion. The seven independent terminal hydrogen atoms were represented by well-shaped peaks 0.24–0.58 electron/A^3 in height, and the bridge hydrogen by a slightly elongated (parallel with the bond) peak 0.26 electron/A^3 in height. Addition of these atoms to the structure gave $R = 5.7\%$. Correction of a minor program error and two more cycles of refinement gave the final parameters and an R of 5.6%. Addition of a remeasured set of 80l and 82l reflections raised the final R to 5.8%. These reflections had been omitted from calculations at an early stage because of serious errors. As before, the parameters refined were the positional parameters of all atoms except hydrogen, anisotropic thermal parameters for the cesium atom, and an isotropic temperature factor for each of the lighter atoms. All the hydrogen atoms included in the structure were given $B = 0.8$, following a suggestion by Jensen[12] that these atoms have values 2 units smaller than attached carbon (or, presumably, other) atoms. Atomic scattering factors used were those given in ref 10b, with real and imaginary anomalous scattering corrections added in the case of the cesium ion. For all atoms except cesium, the scattering factors for neutral atoms were used. The structure-factor program used was a modification of the block-diagonal version of the Gantzel–Sparks–Trueblood program,[13] which minimizes $\Sigma w(|F_o - F_c|)^2$. The goniostat-setting cards were produced with program GSET4 by Dr. C. T. Prewitt. Fourier and other programs were those of the author.

The final set of atomic parameters is given in Table I; the observed and calculated structure factors are compared in Table II. Figure 2 gives the interatomic distances as well as the atomic numbering scheme. The distances and angles in the tetramethylammonium group are listed in Table III and selected angles within the hydroborate cage in Table IV. The crystal packing is illustrated in Figure 3.

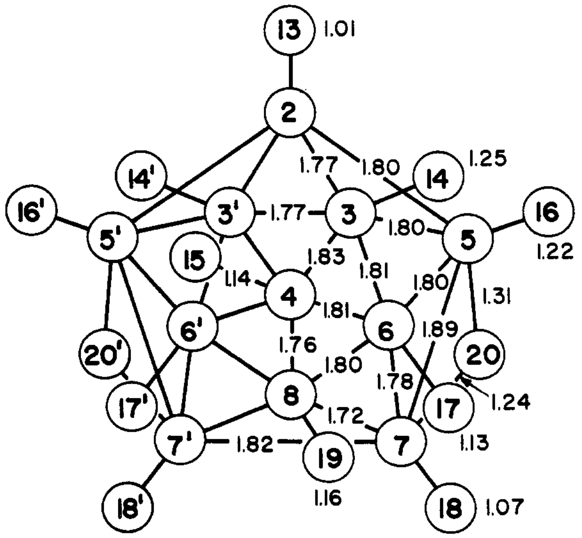

Figure 2.—Schematic molecular structure, with numbering scheme and interatomic distances. The average standard deviation in a B–B bond is 0.013 A, and in a B–H bond it is about 0.1 A.

Discussion

The $B_{11}H_{13}^{2-}$ ion is illustrated in Figure 4. It consists of a boron framework of basically icosahedral symmetry, except that one icosahedral vertex is missing. Each of the eleven boron atoms is bonded to a single terminal hydrogen atom, and the remaining two hydrogen atoms bridge nonadjacent edges of the open pentagonal face.

Because the distance of 1.7 A between the two bridging protons is much shorter than the normal van der Waals contact of 2.4 A,[14] and because these bridging protons appear over the face of the molecule when viewed from above (Figure 5), it is pertinent to compare the location of these atoms relative to the boron skeleton with formally similar fragments in other molecules. If one chooses the reference fragment to be a B_6 pentagonal pyramid with at least one bridged basal edge, *e.g.*, the pyramid having apex B(6) and one edge B(5)–B(7) in $B_{11}H_{13}^{2-}$, either the dihedral angle B(6)B(5)-

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(12) L. H. Jensen, Winter Meeting, American Crystallographic Association, Suffern, N. Y., Feb 24–26, 1965, Paper A-6.

(13) "World List of Crystallographic Computer Programs," 1st ed, International Union of Crystallography, Sept 1962. Program No. 384.

(14) L. Pauling, "The Nature of the Chemical Bond," 2nd ed, Cornell University Press, Ithaca, N. Y., 1948, p 189.

TABLE II

OBSERVED AND CALCULATED STRUCTURE FACTORS[a]

		0	
0	8	218	244
0	10	342	-355
0	12	298	303
0	14	149	-164
0	16	223	227
0	18	264	-263
0	20	169	182
0	22	102	-102
0	24	105	106
0	26	104	-109
0	28	104	95
0	30	74	-65
0	32	26	24
0	34	43	-35
2	6	305	290
2	8	429	-439
2	10	345	359
2	12	204	-208
2	14	242	238
2	16	204	-207
2	18	146	155
2	20	160	-163
2	22	141	142
2	24	106	-103
2	26	97	93
2	28	76	-75
2	30	64	63
2	32	72	-64
2	34	50	46
4	0	320	330
4	2	388	-354
4	4	325	304
4	6	331	-322
4	8	265	250
4	10	179	-177
4	12	302	306
4	14	268	-263
4	16	170	167
4	18	141	-142
4	20	108	110
4	22	123	-125
4	24	118	117
4	26	87	-82
4	28	78	72
4	30	63	-64
4	32	61	56
6	0	284	-251
6	2	268	260
6	4	317	-308
6	6	280	271
6	8	221	-213
6	10	227	217
6	12	170	-165
6	14	153	154
6	16	171	-172
6	18	151	151
6	20	136	-139
6	22	130	125
6	24	103	-97
6	26	60	61
6	28	45	-45
6	30	54	48
6	32	41	-41
8	0	307	300
8	2	213	-205
8	4	153	144
8	6	183	-177
8	8	207	203
8	10	216	-216
8	12	166	167
8	14	137	-134
8	16	131	128
8	18	120	-120
8	20	101	100
8	22	55	-55
8	24	55	56
8	26	90	-83
8	28	78	74
8	30	45	-46
10	0	98	-97
10	2	170	162
10	4	173	-172
10	6	132	137
10	8	167	-165
10	10	135	135
10	12	107	-104
10	14	102	104
10	16	95	-96
10	18	91	89
10	20	88	-80
10	22	81	74
10	24	61	-58
10	26	50	44
12	0	136	137
12	2	112	-113
12	4	122	115
12	6	126	-125
12	8	61	63
12	10	57	-56
12	12	109	110
12	14	103	-99
12	16	74	73
12	18	66	-63
12	20	55	50
12	22	62	-57
14	0	65	-46
14	2	63	70
14	4	88	-90
14	6	82	75
14	8	82	-83
14	10	87	83
14	12	53	-54
14	14	60	57
14	16	55	-53
16	0	73	77
16	2	64	-58
16	4	41	37
16	6	44	37
		1	
1	1	-6	-15
1	2	79	-100
1	3	89	80
1	4	164	139
1	5	37	-35
1	6	22	21
1	7	-9	-3
1	8	-9	-2
1	9	20	-15
1	10	22	-19
1	11	27	-24
1	12	43	-40
1	13	-10	3
1	14	15	8
1	15	-11	-12
1	16	-11	-13
1	17	15	20
1	18	-12	7
1	19	21	20
1	20	-13	17
1	21	-13	-7
1	22	-12	-7
1	23	-13	0
3	1	21	24
3	2	82	73
3	3	51	-40
3	4	17	26
3	5	38	-37
3	6	75	-63
3	7	66	55
3	8	27	16
3	9	16	13
3	10	37	22
3	11	31	-26
3	12	70	-63
3	13	29	24
3	14	15	11
3	15	-12	6
3	16	61	63
3	17	29	-30
3	18	-13	-5
3	19	-13	-2
3	20	36	-35
3	21	18	12
3	22	-13	-1
5	1	11	18
5	2	60	62
5	3	49	-39
5	4	-10	7
5	5	30	-23
5	6	32	-23
5	7	20	17
5	8	32	-28
5	9	34	28
5	10	-11	-1
5	11	-11	2
5	12	30	-26
5	13	21	20
5	14	15	11
5	15	-13	7
5	16	78	78
5	17	37	-38
5	18	-13	-7
5	19	-13	-11
5	20	39	-37
5	21	-13	10
7	1	-11	13
7	2	-11	12
7	3	-11	2
7	4	27	29
7	5	14	-14
7	6	35	33
7	7	30	-26
7	8	53	-49
7	9	13	11
7	10	31	-30
7	11	21	19
7	12	-13	3
7	13	-13	2
7	14	-13	5
7	15	15	4
7	16	36	37
7	17	-13	-15
7	18	-13	-2
9	1	-13	10
9	2	16	-5
9	3	19	14
9	4	29	30
9	5	19	-9
9	6	34	26
9	7	27	-22
9	8	22	-22
9	9	-13	1
9	10	-13	-17
9	11	-13	3
9	12	-13	-4
9	13	-13	1
11	2	-13	6
11	3	-13	-2
		2	
0	5	277	287
0	6	109	-113
0	7	323	-328
0	8	176	184
0	9	253	274
0	10	153	-166
0	11	180	-200
0	12	82	114
0	13	261	275
0	14	127	-145
0	15	211	-215
0	16	102	111
0	17	114	128
0	18	45	-60
0	19	95	-100
0	20	66	77
0	21	119	122
0	22	61	-70
0	23	112	-116
0	24	44	46
0	25	85	87
0	26	43	-47
0	27	81	-81
0	28	34	41
0	29	55	56
0	30	34	-41
0	31	58	-52
0	32	38	39
0	33	59	56
0	34	22	-17
2	3	306	277
2	4	137	-132
2	5	307	-303
2	6	230	230
2	7	276	276
2	8	135	-136
2	9	244	-239
2	10	116	120
2	11	238	242
2	12	157	-159
2	13	183	-192
2	14	97	103
2	15	154	165
2	16	80	-86
2	17	175	-182
2	18	96	100
2	19	153	160
2	20	73	-76
2	21	109	-114
2	22	67	69
2	23	101	103
2	24	59	-62
2	25	87	-87
2	26	42	44
2	27	63	61
2	28	40	-36
2	29	62	-60
2	30	30	27
2	31	50	51
2	32	20	-19
2	33	38	-33
2	34	28	22
4	0	174	151
4	1	368	340
4	2	204	-195
4	3	228	-223
4	4	189	182
4	5	314	296
4	6	116	-109
4	7	227	-222
4	8	151	147
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0	8	60	-68
0	9	90	89
0	10	52	45
0	11	131	-128
0	12	100	-113
0	13	155	150
0	14	69	83
0	15	122	-120
0	16	49	-54
0	17	111	101
0	18	71	73
0	19	89	-84
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0	21	82	74
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0	23	77	-77
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0	27	42	-38
0	28	36	-25
0	29	48	37
0	30	-37	25
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2	1	148	-127
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2	3	197	177
2	4	116	111
2	5	182	-163
2	6	83	-82
2	7	169	156
2	8	112	112
2	9	179	-168
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2	11	137	131
2	12	51	55
2	13	111	-104
2	14	70	-67
2	15	112	104
2	16	66	66
2	17	90	-91
2	18	62	-46
2	19	87	81
2	20	62	57
2	21	94	-78
2	22	47	-46
2	23	50	64
2	24	-36	28
2	25	101	-53
2	26	-38	-32
2	27	50	43
2	28	-36	26
2	29	-36	-36
4	0	135	-126
4	1	193	-183
4	2	84	78
4	3	137	127
4	4	74	-74
4	5	99	-100
4	6	96	94
4	7	143	135
4	8	72	-67
4	9	153	-149
4	10	85	83
4	11	128	128
4	12	79	-79
4	13	113	-109
4	14	59	64
4	15	96	94
4	16	55	-63
4	17	80	-89
4	18	46	47
4	19	73	84
4	20	25	-38
4	21	52	-60
4	22	23	28
4	23	29	38
4	24	25	-33
4	25	43	-48
4	26	25	37
4	27	46	59
4	28	24	-25
6	0	71	71
6	1	128	127
6	2	93	-90
6	3	130	-130
6	4	86	80
6	5	145	139
6	6	71	-70
6	7	118	-116
6	8	80	79
6	9	103	98
6	10	61	-58
6	11	103	-98
6	12	52	50
6	13	76	76
6	14	45	-46
6	15	72	-72
6	16	45	49
6	17	92	91
6	18	50	-57
6	19	78	-79
6	20	43	46
6	21	60	64
6	22	36	-41
6	23	55	-63
6	24	23	30
6	25	34	42
6	26	19	-18
6	27	18	-24
8	0	64	-59
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8	2	73	63
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8	4	64	-63
8	5	109	-104
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8	7	94	93
8	8	54	-54
8	9	82	-82
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10	17	36	50
10	18	16	-25
10	19	29	-44
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12	15	24	37
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14	2	21	-26
14	3	39	-44
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1	2	-14	9
1	3	-14	-16
1	4	-14	-6
1	5	-14	10
1	6	-14	-0
1	7	-14	-35
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1	9	-14	-6
1	10	-14	8
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0	5	117	-124
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0	8	62	64
0	9	129	-139
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0	21	58	-60
0	22	34	-32
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0	24	30	25
0	25	46	-46
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2	7	120	-125
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2	9	88	86
2	10	60	58
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2	19	74	-76
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2	22	27	28
2	23	55	-56
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4	9	103	-105
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4	11	95	93
4	12	53	51
4	13	79	-82
4	14	41	-42
4	15	73	72
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4	17	79	-78
4	18	39	-36
4	19	64	67
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4	21	52	-52
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6	2	56	55
6	3	93	-88
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6	9	99	98
6	10	56	55
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6	18	30	27
6	19	50	-48
6	20	29	-25
6	21	50	48
6	22	20	20
6	23	36	-35
6	24	15	-40
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8	1	87	-86
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8	3	88	85
8	4	43	41
8	5	85	-83
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8	7	65	65
8	8	36	31
8	9	64	-60
8	10	32	-27
8	11	65	64
8	12	43	40
8	13	57	-55
8	14	33	-32
8	15	62	60
8	16	21	23
8	17	61	-58
8	18	27	-25
8	19	45	44
8	20	24	24
8	21	43	-40
10	0	35	-30
10	1	66	66
10	2	32	35
10	3	57	-59
10	4	39	-36
10	5	62	61
10	6	32	29
10	7	76	-66
10	8	39	-29
10	9	52	50
10	10	24	30
10	11	34	-50
10	12	20	-24
10	13	34	54
10	14	-15	23
10	15	23	-39
10	16	-15	-29
12	0	25	24
12	1	41	-41
12	2	26	-23
12	3	52	52
12	4	18	22
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		12	
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0	1	17	-3
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0	4	82	88
0	5	-14	-13
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0	8	97	101
0	9	-14	6
0	10	88	-88
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0	13	-16	-11
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0	19	-15	4
0	20	62	61
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0	22	48	-45
0	23	-15	5
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2	2	108	102
2	3	-13	-9
2	4	96	-92
2	5	15	-1
2	6	87	84
2	7	-13	1
2	8	92	-90
2	9	-13	11
2	10	86	88
2	11	-14	-6
2	12	84	-82
2	13	-14	-1
2	14	87	86
2	15	-14	-5
2	16	67	-66
2	17	14	3
2	18	47	45
2	19	-14	-1
2	20	50	-47
2	21	-15	3
2	22	42	39
4	0	57	55
4	1	14	-7
4	2	85	-77
4	3	-14	-4
4	4	119	113
4	5	-14	-4
4	6	108	-104
4	7	-14	10
4	8	83	76
4	9	-13	-3
4	10	78	-74
4	11	-14	3
4	12	71	71
4	13	-14	-3
4	14	71	-71
4	15	14	-1
4	16	59	60
4	17	-14	-4
4	18	44	-40
4	19	-14	6
4	20	48	45
4	21	-15	-0
6	0	111	-106
6	1	15	5
6	2	93	90
6	3	14	-9
6	4	75	-72
6	5	-14	-1
6	6	73	72
6	7	17	2
6	8	61	-57
6	9	-14	7
6	10	55	51
6	11	-15	-4
6	12	73	-68
6	13	-15	-1
6	14	66	62
6	15	-15	-4
6	16	57	-54
6	17	-15	4
6	18	56	53
6	19	-15	-1
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8	1	-14	-1
8	2	67	-63
8	3	-14	1
8	4	59	57
8	5	17	-9
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8	7	-15	7
8	8	65	61
8	9	-15	3
8	10	77	-75
8	11	-15	2
8	12	49	46
8	13	-15	-8
8	14	34	-35
8	15	-14	2
10	0	61	-62
10	1	-15	2
10	2	53	54
10	3	-15	-6
10	4	51	-47
10	5	-14	1
10	6	47	45
10	7	-15	1
10	8	48	-44
		14	
0	0	32	26
0	1	29	39
0	2	25	-35
0	3	48	-61
0	4	26	36
0	5	46	49
0	6	27	-34
0	7	45	-54
0	8	36	44
0	9	40	49
0	10	27	-37
0	11	36	-47
0	12	19	29
0	13	43	51
0	14	25	-34
0	15	38	-51
0	16	22	21
0	17	22	21
2	0	46	-42
2	1	66	-62
2	2	40	38
2	3	58	56
2	4	40	-38
2	5	59	-54
2	6	38	36
2	7	52	50
2	8	28	-24
2	9	44	-44
2	10	26	27
2	11	46	45
2	12	32	-32
2	13	45	-43
2	14	21	23
2	15	38	38
2	16	29	-27
4	0	42	39
4	1	64	62
4	2	38	-35
4	3	55	-48
4	4	34	29
4	5	45	40
4	6	33	-32
4	7	53	-51
4	8	39	36
4	9	55	52
4	10	31	-29
4	11	42	-43
4	12	24	23
4	13	38	33
4	14	27	-24
6	0	24	-24
6	1	47	-40
6	2	30	28
6	3	49	45
6	4	34	-31
6	5	48	-42
6	6	33	27
6	7	45	42
6	8	34	-27
6	9	55	-49
6	10	36	29
6	11	44	41
8	0	30	-6
		16	
0	0	29	-24
0	1	-19	52
0	2	-16	24
0	3	-18	21

[a] Each group of four columns contains *k*, *l*, $|F_o|$, and $|F_c|$ and is headed by the common value of *h*. Because of the complex scattering factor for Cs^+, the phase in each case can differ from 0 or 180°; it does so by more than about 7° only for very small reflections. The sign in the $|F_o|$ column thus indicates the approximate phase. Negative values in the $|F_o|$ column are threshold values for unobserved reflections.

TABLE III

GEOMETRY OF THE TETRAMETHYLAMMONIUM ION[a]

Atoms			Distance, A	Angle, deg
i	j	k	i–j	i–j–k
C(10)	N(9)	C(11)	1.52 (3)	114 (3)
C(10)	N(9)	C(12)	. . .	108 (3)
C(11)	N(9)	C(12)	1.48 (3)	111 (3)
C(12)	N(9)	C(12′)	1.54 (2)	105 (2)

[a] Figures in parentheses are standard deviations applicable to the final figure tabulated.

B(7)–B(5)B(7)H(20) or the distance of the bridge hydrogen from the B(6)B(5)B(7) plane may be used as comparison. The proton H(20) is displaced 0.1 A from this plane in $B_{11}H_{13}^{2-}$, toward the open face, giving a dihedral angle of 9°. In B_6H_{10}[9] (Figure 1) H(5) is displaced by 0.56 A and H(6) by 0.62 A, again inward toward the bases of their respective reference pentagonal pyramids. The displacements of the bridge hydrogens H(6) and H(7) in $B_{10}H_{14}$[15] are, respectively, 0.19 and 0.36 A. Thus the inward tilt of the bridges in $B_{11}H_{13}^{2-}$ is considerably less than in at least these two cases, and these must be considered "normal" bridges.

Although the structure of $B_{11}H_{13}^{2-}$ does not com-

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[15] E. B. Moore, Jr., R. E. Dickerson, and W. N. Lipscomb, *J. Chem. Phys.*, **27**, 209 (1957).

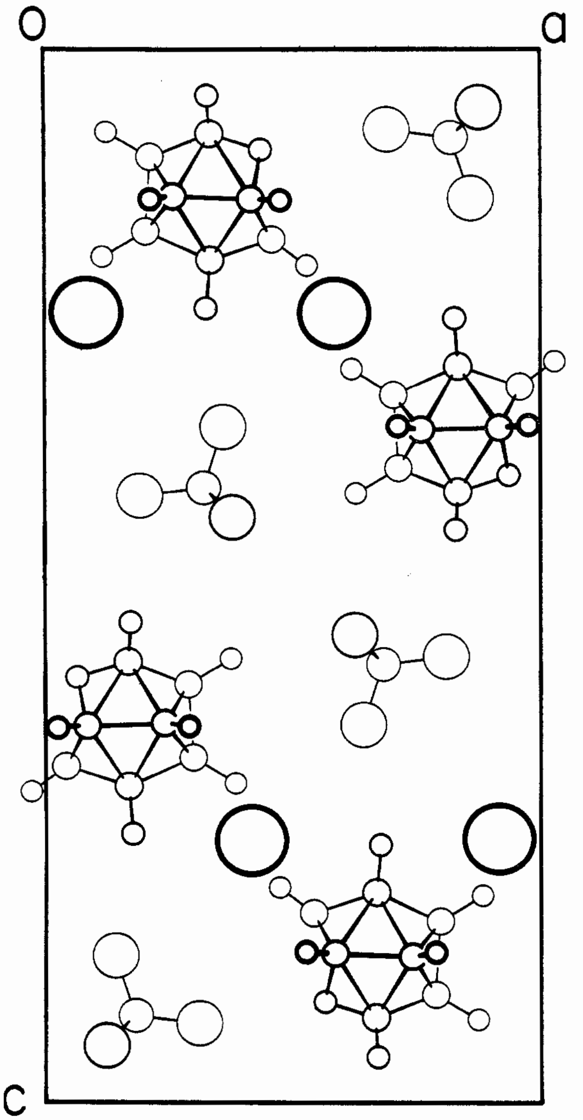

Figure 3.—Packing in $CsN(CH_3)_4B_{11}H_{13}$. Only one layer of molecules is shown. The next layer can be produced by the C-centering operation.

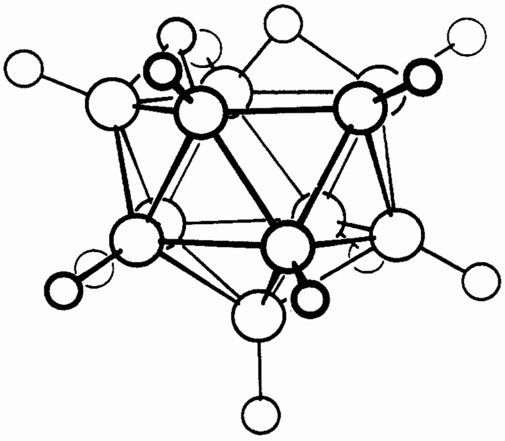

Figure 4.—The $B_{11}H_{13}^{2-}$ ion.

TABLE IV

SELECTED ANGLES WITHIN THE HYDROBORATE ANION

Atoms			Angle i–j–k, deg[a]	Atoms			Angle i–j–k, deg[a]
i	j	k		i	j	k	
B(5′)	B(2)	B(5)	108.4	B(3)	B(5)	H(16)	122
B(2)	B(5)	B(7)	108.9	B(6)	B(5)	H(16)	124
B(5)	B(7)	B(7′)	106.9	B(7)	B(5)	H(16)	123
B(3′)	B(3)	B(6)	108.9	B(3)	B(6)	H(17)	117
B(3)	B(6)	B(8)	106.3	B(4)	B(6)	H(17)	122
B(6)	B(8)	B(6′)	109.6	B(8)	B(6)	H(17)	129
B(3)	B(2)	H(13)	120	B(7)	B(6)	H(17)	123
B(5)	B(2)	H(13)	122	B(5)	B(6)	H(17)	118
B(2)	B(3)	H(14)	120	B(5)	B(7)	H(18)	126
B(3′)	B(3)	H(14)	114	B(6)	B(7)	H(18)	122
B(4)	B(3)	H(14)	117	B(8)	B(7)	H(18)	120
B(6)	B(3)	H(14)	126	B(7′)	B(7)	H(18)	120
B(5)	B(3)	H(14)	128	B(4)	B(8)	H(19)	116
B(3)	B(4)	H(15)	122	B(6)	B(8)	H(19)	120
B(6)	B(4)	H(15)	122	B(7)	B(8)	H(19)	121
B(8)	B(4)	H(15)	123	B(5)	B(7)	H(20)	44
B(2)	B(5)	H(16)	120	B(7)	B(5)	H(20)	41

[a] Standard deviations are about 0.9° for B–B–B angles and 4° for B–B–H angles.

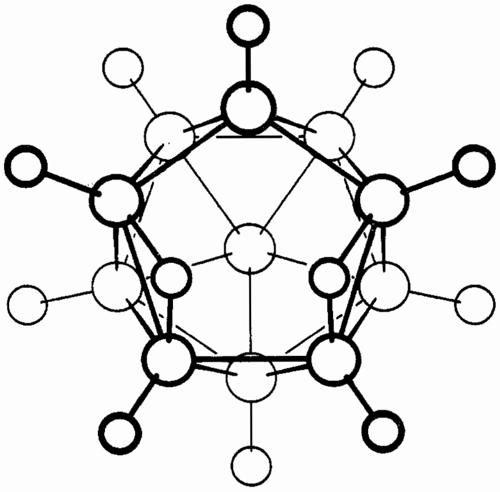

Figure 5.—View of $B_{11}H_{13}^{2-}$ along its fivefold axis.

pletely exclude the possibility that in $B_{11}H_{14}^-$ there is H···H interaction so that none of the exchanging protons is strictly part of a BH_2 group, it does seem likely that structures II are appropriate representations of $B_{11}H_{14}^-$. In this case, of course, only one of the two protons in the BH_2 group must participate in the rapid exchange and the other remain essentially a terminal proton.

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