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Molecular and Crystal Structure of Dimethyl-1,6-dicarbaclovononaborane(9)

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The molecule of $B_7H_7C_2(CH_3)_2$, dimethyl-1,6-dicarbaclovononaborane(9), has C_{2v} symmetry. Distortions of the B_7C_2 unit are only slight from an idealized B_9 polyhedron based upon a tricapped prism of D_{3h} symmetry. The C atoms, with attached CH_3 groups, are at two of the three apices of the tricapped prism. Cumulative charges in the six highest filled molecular orbitals give in order of increasing charge B_8 (attached to no C atom), B_4 (B_5, B_7, B_9) (attached to one C), and B_2 (B_3) (attached to two C atoms). The crystal structure as determined at −40° is of symmetry $P2_1/a$ with eight molecules in the unit cell of parameters $a = 13.59$, $b = 11.11$, $c = 12.47$ Å, and $\beta = 93.19°$. The 1883 observed X-ray reflections were refined to $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.04$.

As part of a series on structures and charge distribution in carboranes and substituted carboranes, we report X-ray diffraction and molecular orbital studies on $B_7H_7C_2(CH_3)_2$. This molecule is obtained,[1] along with $B_6H_6C_2(CH_3)_2$ and one isomer of $B_8H_8C_2(CH_3)_2$ when $B_7C_2H_{11}(CH_3)_2$ is pyrolyzed near 200° in diphenyl ether as solvent. Three types of B atoms, in the ratio 1:2:4, appear in the ^{11}B nuclear magnetic resonance spectrum[1] at 19.3 Mc. In accord with the $B_{10}H_{10}^{2-}$ assignment,[2] the single B atom at lowest field was assigned[1] to an apical position, and all others were assigned to the trigonal-prism part of a tricapped prism having two C atoms at the other apices. We confirm this structure, also indicated in our preliminary report,[3] and give geometrical details of the molecule in the present study.

Experimental Section

Liquid $B_7H_7C_2(CH_3)_2$ (mp −21 to −22°) was vacuum distilled into Pyrex capillaries having uniform wall thickness in amounts which yielded crystals, when grown in a stream of N_2,[4] about 0.5–1.0 mm in all dimensions. All X-ray data were taken at a sample temperature of −30 to −45°, but repeated growth of new crystals was required at about 2-week intervals because of thermal shock during filling of the low-temperature dewar with liquid N_2. Reciprocal lattice symmetry of C_{2h} and systematic absences of $h0l$ for h odd and $0k0$ for k odd indicated that the space group is $P2_1/a$. Unit cell parameters of $a = 13.592 \pm 0.005$, $b = 11.109 \pm 0.008$, $c = 12.468 \pm 0.003$ Å, and $\beta = 93.19 \pm 0.03°$ were obtained from Weissenberg photographs which had been calibrated by superposition of a powder diffraction pattern of Al. A least-squares procedure minimizing

$$\sum_{n=1}^{N} w_n(\sin^2 \theta_{\text{obsd}} - \sin^2 \theta_{\text{calcd}})^2(4/\lambda_n^2)^2$$

was used, where the weights $w_n = 1/\sin^2 \theta_{\text{obsd}}$ and $N = 52$ reflections. A reasonable density of 0.967 g/cm³ was calculated from the assumption of eight molecules in the unit cell, but no observed density is available for comparison. The related but somewhat less dense compound[5] $B_7C_2H_{11}(CH_3)_2$ has a density of 0.92 g/cm³. There are thus two molecules in the asymmetric unit, and no molecular symmetry is demanded by the space group.

Precession data were taken with Cu Kα (Ni filter) and Mo Kα (Zr filter) radiation, and Weissenberg data were collected with Cu Kα (Ni filter) radiation. The 21 levels included Hkl for $0 \leqslant H \leqslant 3$; hKl for $0 \leqslant K \leqslant 8$ except $K = 3$; $hk0$; [101] for levels 0, 1, 2, and 3; [111] for levels 0, 1, and 2; and [110] for level 0 only. It need hardly be added that the crystal grew with no preferred orientation. Of the 1883 distinct reflections (Table I) representing 42% of the Cu Kα sphere, some 446 were below the observational limit on the films. Intensities on Weissenberg films were measured with an intensity scale prepared from the same substance, and intensities on precession films were recorded on a twin-beam microdensitometer. Corrections were applied for Lorentz and polarization factors and for oblique incidence of X-rays on the higher level films. Scale factors for the appropriate sets of data were calculated by least-squares procedures which minimize[6]

$$R = \sum_{\substack{hij \\ i \neq j}} w_{hij}(\ln (s_i I_{hi}) - \ln (s_j I_{hj}))^2$$

where s_i is the scale factor for the ith set, I_{hi} is the measurement of reflection h on set i, $w_{hij} = 1/(f_{hi}^2 + f_{hj}^2)$, and f_{hi} is the estimated relative error in I_{hi}. Reflections were then assigned weights according to the function

$$w_h = \sqrt{f_{hi}}\sqrt{f_{hj}}\left(\frac{1}{s_i I_{hi}} \frac{1}{s_j I_{hj}}\right)$$

A calculation of μR from $\mu = 2.87$ cm⁻¹ for Cu Kα radiation (0.46 cm⁻¹ for Mo Kα radiation) for $R = 1$ mm for our largest crystal indicates that absorption corrections are less than 4%.

Structure Determination and Refinement

The structure was solved by application of Sayre's method[7] with the use of a modified form of Long's program[8] (REL) for the IBM 7094. First, the normalized structure factors

$$E_{hkl}^2 = F_{hkl}^2[\epsilon \sum_{i=1}^{N} f_i^2(hkl)]^{-1}$$

were calculated for all reflections for which $\rho = (\sin^2 \theta)/\lambda^2 \leqslant 0.35$, where F_{hkl}^2 has been corrected for thermal motion, $\epsilon = 2$ for $0k0$ and $h0l$, and $\epsilon = 1$ for all other reflections in the $P2_1/a$ space group. The sum over[9] f_i^2 extends over the $N = 192$ atoms in the unit cell

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[1] F. N. Tebbe, P. M. Garrett, D. C. Young, and M. F. Hawthorne, *J. Am. Chem. Soc.*, **88**, 609 (1966).

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[4] W. N. Lipscomb, *Norelco Reptr.*, **4**, 56 (1957).

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[6] (a) P. Simpson, Ph.D. Thesis, Harvard University, 1963; (b) A. D. Rae, *Acta Cryst.*, **19**, 683 (1965); (c) A. D. Rae and A. B. Blake, *ibid.*, **20**, 586 (1966).

[7] D. Sayre, *ibid.*, **5**, 60 (1952).

[8] R. E. Long, Ph.D. Thesis, Part III, UCLA, 1965.

[9] All f_i values were obtained from the "International Tables for X-ray Crystallography," Vol. 3, The Kynoch Press, Birmingham, England, 1962, p 202.

TABLE I

COMPARISON OF OBSERVED AND CALCULATED STRUCTURE FACTORS FOR $B_7H_7C_2(CH_3)_2$[a,b]

H	L	FO	FC
K = 12			
1	2	49	56
-1	2	47	54
0	2	47	-55
1	1	28	-28
-1	1	-31	5
1	0	66	-78
0	0	112	128
K = 11			
1	5	48	-50
2	4	62	84
1	4	-31	9
-1	4	46	-35
2	3	-41	-43
1	3	62	72
-1	3	32	22
2	2	58	-61
1	2	31	2
-1	2	-31	1
0	2	-45	-23
2	1	-40	-30
1	1	54	-60
-1	1	31	11
2	0	-40	15
1	0	43	48
K = 10			
-1	8	44	-54
-1	7	41	-71
1	6	56	-54
-1	6	-31	18
2	5	40	43
1	5	31	31
-1	5	-31	-27
2	4	-41	32
1	4	60	61
-1	4	55	-63
10	3	31	30
2	3	73	-71
1	3	33	-33
-1	3	74	72
10	2	-25	-12
2	2	-41	36
1	2	-32	-12
-1	2	67	76
0	2	49	-54
10	1	-25	14
2	1	50	56
1	1	-31	-11
-1	1	36	-41
10	0	-26	29
2	0	-41	34
1	0	-31	-7
0	0	47	42
K = 9			
0	10	52	29
2	7	40	-40
1	7	48	-50
9	6	-26	-18
-9	6	23	18
3	6	64	-70
2	6	43	46
1	6	42	42
-1	6	44	38
0	6	46	-56
9	5	51	-46
3	5	-25	1
2	5	-41	18
-2	5	92	-116
1	5	-31	-11
-1	5	31	3
0	5	95	-109
9	4	-27	-15
-9	4	-27	-12
3	4	47	51
-3	4	80	97
2	4	128	-154
-2	4	72	-91
1	4	98	-94
-1	4	-31	-19
0	4	81	82
-9	3	69	-63
3	3	57	-68
-3	3	115	-122
2	3	95	95
-2	3	-41	-20
1	3	36	-30
-1	3	-31	-19
9	2	-27	6
-9	2	-27	27
3	2	-25	-8
-3	2	34	-38
2	2	-40	-31
-2	2	-40	-7
1	2	47	54
-1	2	99	-112
0	2	45	-16
9	1	-27	-24
-9	1	-27	23
3	1	89	105
-3	1	56	65
2	1	141	-177
-2	1	56	-65
1	1	52	-54
-1	1	65	73
0	1	45	43
9	0	-27	1
4	0	182	173
3	0	63*	-51
2	0	-40	14
1	0	77	74
K = 8			
3	12	35	-37
2	12	121	86
1	12	-44	-2
0	12	67	-54
0	11	-52	1
9	10	-36	46
7	10	-43	15
6	10	-47	13
-6	10	67	69
5	10	-51	27
-5	10	56	-41
4	10	-53	-33
-4	10	130	-124
3	10	-55	-1
-3	10	-51	5
2	10	-56	8
-2	10	68	-9
1	10	-57	15
-1	10	-52	9
0	10	74	75
9	9	91	-80
8	9	57*	44
-8	9	-26	32
7	9	84	61
6	9	-53	-21
5	9	67	-46
4	9	76	53
3	9	-59	2
2	9	-59	23
-2	9	-41	-18
1	9	45	44
0	9	56	55
-9	8	40	37
8	8	61*	-46
-8	8	57	63
7	8	91	-63
-7	8	51	-44
6	8	89	56
-6	8	78	-70
5	8	67	61
-5	8	-55	26
4	8	-60	-26
-4	8	-55	-36
3	8	56	33
-3	8	-55	24
2	8	78*	-62
-2	8	59	57
1	8	-31	-21
-1	8	44	37
0	8	-59	-2
11	7	48	45
10	7	95	-65
-10	7	41	31
9	7	-52	-13
-9	7	-50	17
8	7	-55	9
-8	7	-27	4
7	7	-58	-21
-7	7	-54	-15
6	7	-59	-11
-6	7	-55	-43
5	7	-60	14
-5	7	-55	-18
4	7	71	-61
-4	7	-53	-36
3	7	79*	-61
-3	7	48	50
2	7	43	-35
-2	7	42	32
1	7	32	29
-1	7	71*	-79
0	7	84*	84
11	6	56	34
-11	6	77	47
10	6	-51	-17
-10	6	96	-72
9	6	-55	16
-9	6	43	-30
8	6	58*	-54
-8	6	-27	-17
7	6	78	39
-7	6	49	37
6	6	-60	23
-6	6	44	-39
5	6	48	-41
-5	6	84	61
4	6	-55	28
-4	6	143	133
3	6	-25	-13
-3	6	49	-51
2	6	95	105
-2	6	120	135
1	6	60*	34
-1	6	-31	-1
0	6	57	-60
12	5	35	-17
11	5	-49	-23
-11	5	85	-74
10	5	-54	2
-10	5	123	80
9	5	51	40
-9	5	-54	30
8	5	89*	-42
-8	5	40	-48
7	5	-60	-21
-7	5	49	-39
6	5	-57	-36
-6	5	116	83
5	5	-53	30
-5	5	-46	-0
4	5	210	173
-4	5	73	60
3	5	41*	29
-3	5	-29	24
2	5	165	-160
-2	5	139	162
1	5	-31	-22
-1	5	-31	10
0	5	-41	20
12	4	57	-44
11	4	62	-43
10	4	101	72
9	4	67	-52
8	4	-27	3
-8	4	57*	41
7	4	-58	-19
-7	4	66	-29
6	4	127	-75
-6	4	59	35
5	4	70	36
-5	4	42	32
4	4	45	-31
-4	4	56	41
3	4	50	-51
-3	4	31	16
2	4	73	70
-2	4	125	-132
1	4	33	30
-1	4	57	54
0	4	-36	-25
13	3	91	-65
12	3	138	104
-12	3	58	52
11	3	53	39
-11	3	64	39
10	3	-58	-12
-10	3	-54	-23
9	3	-60	-0
-9	3	34	-29
8	3	-27	28
-8	3	62*	-56
7	3	133	-92
-7	3	-48	18
6	3	82	-70
-6	3	-43	-30
5	3	50	47
-5	3	141	127
4	3	62	-69
-4	3	48	45
3	3	50*	-43
-3	3	34	-41
2	3	172	184
-2	3	108	-106
1	3	79	97
-1	3	-29	-8
0	3	157*	-194
12	2	86	-78
11	2	-56	-14
9	2	-60	29
8	2	62*	-54
-8	2	101*	63
7	2	-52	-4
-7	2	73	60
6	2	49	17
-6	2	-41	19
5	2	188	-135
-5	2	54	-50
4	2	136	-114
-4	2	57	44
3	2	38*	49
-3	2	42	-33
2	2	114	-125
-2	2	40	35
1	2	118*	-126
-1	2	60	-64
0	2	125*	156
13	1	68	62
12	1	62	-39
11	1	55	-50
10	1	-59	-31
9	1	93	73
-9	1	75	56
8	1	81*	79
-8	1	-28	23
7	1	78	57
-7	1	-45	3
6	1	239	-151
-6	1	341	269
5	1	42	30
-5	1	-34	-7
4	1	138	-138
-4	1	89	-53
3	1	125	-131
-3	1	185	-189
2	1	44*	-45
-2	1	25	16
1	1	47	-56
-1	1	74*	105
0	1	-18	26
-12	0	70	45
-11	0	59	-62
-10	0	-54	-20
-9	0	54	-27
8	0	-28	15
-8	0	-51	15
-7	0	42	-23
6	0	-54	-15
-6	0	43	-15
5	0	107	114
-5	0	156	-114
4	0	-57	17
-4	0	-32	17
3	0	-23	-17
-3	0	-27	17
2	0	120*	-117
-2	0	111	-117
1	0	-28	1
-1	0	-16	-1
0	0	103	-104
K = 7			
7	11	53	-53
1	11	47	-43
7	10	51	56
-7	10	-27	16
6	10	49	-51
5	10	-40	-40
4	10	-41	-18
3	10	-42	46
2	10	62	63
-2	10	70	-64
1	10	-31	20
0	10	-45	24
7	9	-27	25
-7	9	26	25
6	9	56	46
5	9	-42	-14
4	9	35	-25
3	9	-42	0
2	9	51	45
-2	9	45	45
1	9	50	53
-1	9	44	43
8	8	70	50
7	8	-27	-12
-7	8	-27	7
6	8	84	70
5	8	-41	17
4	8	-40	-10
3	8	47*	-52
2	8	38	-24
-2	8	-41	-30
1	8	42	46
-1	8	48	51
0	8	67	71
9	7	85	-58
8	7	58	-46
7	7	67	37
-7	7	-27	-9
6	7	-41	7
5	7	-38	15
4	7	111	-87
3	7	32	-33
2	7	52	51
-2	7	163	-189
1	7	33	-35
-1	7	46*	55
0	7	88	-93
-9	6	61	-51
7	6	-28	-14
-7	6	-28	2
-6	6	-31	16
-5	6	41	-42
4	6	74	67
-4	6	181	-202
3	6	56*	42
-3	6	29	35
2	6	39	-45
-2	6	44	41
1	6	99	-102
-1	6	26	19
0	6	-30	-2
-12	5	235	-201
-11	5	-35	8
-10	5	-37	-16
-9	5	50	57
-8	5	-34	-23
7	5	-28	8
-7	5	33	-34
6	5	118	102
-6	5	57	-63
9	5	-33	4
-5	5	32	34
4	5	-31	11
-4	5	27	-30
3	5	40	39
-3	5	92	82
2	5	77	-74
-2	5	-25	-27
1	5	28	21
-1	5	68	-79
0	5	296	343
12	4	58	48
-12	4	33	-37
11	4	55	67
-11	4	53	53
10	4	100	-79
-10	4	-37	-23
9	4	55	-61
-9	4	47	-56
8	4	49	-41
-8	4	83	84
7	4	56*	-51
-7	4	58	-60
6	4	61	43
-6	4	72	-83
5	4	61	53
-5	4	97	70
4	4	-29	-14
-4	4	153	148
3	4	78	-77
-3	4	33	-37
2	4	122	141
-2	4	179	192
1	4	29	-24
-1	4	23	27
0	4	24	-30
-13	3	55	6
12	3	58	-42
11	3	-41	2
10	3	-42	36
-10	3	135	112
9	3	-40	14
-9	3	47	-49
8	3	143	-122
-8	3	44	-46
7	3	39	-40
-7	3	48	33
6	3	86	50
-6	3	99	-87
5	3	64	-56
-5	3	-25	-28
4	3	205	183
-4	3	117	107
3	3	109	114
-3	3	82	90
2	3	137	-140
-2	3	102	116
1	3	-22	-24
-1	3	-18	10
0	3	-21	4
12	2	58	-46
-12	2	78	69
11	2	-42	-23
-11	2	33	-27
10	2	54	47
-10	2	-36	1
9	2	-38	16
-9	2	30	2
8	2	34	-24
-8	2	189	-172
7	2	-27	-18
-7	2	-28	-4
6	2	76	-56
-6	2	126	107
5	2	73	-69
-5	2	120	110
4	2	70	49
-4	2	114	-96
3	2	85	90
-3	2	22	19
2	2	20	-17
-2	2	99	-57
1	2	67	79
-1	2	38	-32
0	2	-17	-4
14	1	52	53
13	1	64	-53
-13	1	63	60
12	1	33	20
-12	1	-35	4
11	1	-43	-14
-11	1	59	37
10	1	52	-50
-10	1	-35	9
9	1	-37	4
-9	1	-32	8
8	1	70	70
-8	1	84	-71
7	1	66	-50
-7	1	88	76
6	1	333	-288
-6	1	220	-161
5	1	32	-23
-5	1	74	59
4	1	194	152
-4	1	174	149
3	1	151	-154
-3	1	114	-95
2	1	82	74
-2	1	-15	21
1	1	65	63
-1	1	34	-44
0	1	34*	-26
-11	0	-37	-2
-10	0	58	-57
-9	0	54	50
8	0	189	-180
-8	0	238	180
-7	0	78	80
6	0	-97	43
-6	0	52	-43
5	0	84	-76
-5	0	87	-76
4	0	214	-203
-4	0	282	203
3	0	88	-84
-3	0	117	-84
2	0	-34	-21
-2	0	32	22
1	0	-26	-7
-1	0	-10	-7
K = 6			
-4	12	44	-35
-3	12	-28	23
2	12	126	-86
-2	12	-29	4
1	12	31	-24
-1	12	-29	13
0	12	42	24
6	11	-26	3
-6	11	-27	8
-5	11	29	-10
-4	11	-30	-16
3	11	79	60
-3	11	76	67
2	11	65	-53
-2	11	-30	16
1	11	60	-56
-1	11	-30	-25
0	11	41	-43
-9	10	40	-38
-8	10	46	33
-7	10	-30	-4
-6	10	46	-52
-5	10	-30	13
4	10	61	49
-4	10	59	55
3	10	-33	11
-3	10	-29	-23
2	10	-32	24
-2	10	-29	19
1	10	-31	7
-1	10	91	-98
0	10	66*	-61
-10	9	36	-31
-9	9	-29	-17
-8	9	61	-62
-7	9	-30	-13
6	9	48	-63
-6	9	-27	3
5	9	32	23
-5	9	37	39
4	9	-31	-0
-4	9	-27	5
3	9	47	40
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1	2	-14	14
-1	2	43	-54
0	2	172	155
-6	1	73	79
-5	1	-36	-2
4	1	-39	-24
-4	1	29	20
3	1	167	172
-3	1	148	-77
2	1	138	-133
-2	1	88	-69
1	1	314*	234
-1	1	51	53
0	1	40	-26
11	0	177	185
10	0	98	-86
9	0	152	-128
8	0	-57	33
7	0	214	-193
6	0	-53	-42
5	0	133	112
4	0	77	-29
3	0	301	-263
-3	0	279	-263
2	0	144	-119
-2	0	156	119
1	0	143	-105
K = 2			
-2	15	23	-20
2	14	30	-31
-2	14	-27	-20
-2	13	61	51
2	12	-27	31
1	12	43	38
2	11	-28	-5
-2	11	-28	8
1	11	-31	-11
2	10	62	-59
1	10	95	-73
0	10	86	66
3	9	145	-121
-3	9	80	-92
2	9	139	132
-2	9	124	132
1	9	101	89
-1	9	93	-94
0	9	78	-68
3	8	134	128
-3	8	72	79
2	8	-24	13
-2	8	30	19
1	8	28	24
-1	8	128	127
0	8	77	-71
-5	7	-40	-3
-4	7	43	-21
3	7	146	125
-3	7	65	53
2	7	93	75
-2	7	85	-80
1	7	118	-91
-1	7	94	99
0	7	36	34
-6	6	-43	-32
5	6	-44	28
-5	6	187	193
4	6	-46	-13
-4	6	68	65
3	6	160	-136
-3	6	111	103
2	6	148	134
-2	6	21	-6
1	6	270	249
-1	6	110	-98
0	6	95	78
-7	5	52	58
4	5	-45	3
-6	5	129	110
5	5	79	66
-5	5	-45	-10
4	5	127	-100
-4	5	147	150
3	5	79	51
-3	5	71	72
2	5	33	-27
-2	5	61	56
1	5	119	-101
-1	5	20	11
0	5	-29	-25
-8	4	117	100
7	4	127	-128
-7	4	62	90
6	4	132	-101
-6	4	190	214
5	4	-44	-16
-5	4	127	103
4	4	148	-128
-4	4	-19	26
3	4	-15	0
-3	4	133	-122
2	4	28	-30
-2	4	105	92
1	4	164	-113
-1	4	58	-17
0	4	-26	-22
8	3	-42	14
-8	3	116	130
7	3	-46	-39
-7	3	-45	9
6	3	-44	-8
-6	3	172	-177
5	3	230	-171
-5	3	46	59
4	3	191	-158
-4	3	64	-38
3	3	156	86
-3	3	146	-106
2	3	21	0
-2	3	108	-113
1	3	181	137
-1	3	89	-92
0	3	126	85
9	2	213	210
-9	2	-35	-10
8	2	185	-177
-8	2	78	-68
7	2	72	-77
-7	2	-46	-33
6	2	253	214
-6	2	-43	-17
5	2	160	-129
-5	2	110	-103
4	2	137	-113
-4	2	41	27
3	2	227	-151
-3	2	189	-154
2	2	159	-130
-2	2	40	13
1	2	21	34
-1	2	102	-80
0	2	371	355
9	1	81	-88
-9	1	64	35
8	1	-46	-42
-8	1	77	-56
7	1	236	193
-7	1	195	183
6	1	56	98
-6	1	-42	-45
5	1	72	77
-5	1	-36	-7
4	1	218	157
-4	1	149	147
3	1	297*	-301
-3	1	23	-15
2	1	410*	-371
-2	1	53	-34
1	1	341	311
-1	1	494	405
0	1	52	26
11	0	127	-95
10	0	-58	-10
9	0	88	-94
8	0	74	75
7	0	-45	31
6	0	158	-129
5	0	83	68
4	0	62	-47
3	0	-10	-12
2	0	160	-88
-2	0	145	-88
1	0	472	-426
-1	0	435	426
0	0	872	959
K = 1			
1	14	33	-31
-1	14	-27	8
1	13	-27	-13
-1	13	-27	-23
3	12	29	31
1	12	46	-32
-1	12	-27	-11
3	11	70	69
1	11	47*	-32
-1	11	-28	20
0	11	126	136
3	10	25	1
2	10	44	21
1	10	71	79
-1	10	-27	-12
0	10	-45	-27
3	9	68	-60
2	9	178	-159
1	9	27	-26
-1	9	63	-55
0	9	109	-99
3	8	58	34
-3	8	-25	-21
2	8	44	-49
-2	8	94	91
1	8	141	-136
-1	8	81	71
0	8	43	39
4	7	112	102
-4	7	79	81
3	7	63	-52
-3	7	-23	-0
2	7	56	48
-2	7	155	-134
1	7	99	101
-1	7	40	-41
0	7	178	-174
5	6	-40	49
-5	6	-28	-39
4	6	211	208
-4	6	73	87
3	6	55	48
-3	6	172	179
2	6	143	138
-2	6	122	-120
1	6	-19	3
-1	6	104	-84
0	6	-32	-22
-7	5	-38	24
6	5	-41	16
-6	5	76	77
5	5	48	33
-5	5	50	51
4	5	78	-89
-4	5	261	-245
3	5	219	146
-3	5	129	142
2	5	338	280
-2	5	40	-41
1	5	41*	-46
-1	5	127	111
0	5	240	208
7	4	-40	46
-7	4	-28	13
6	4	226	-176
-6	4	120	122
5	4	43	-54
-5	4	119	95
4	4	99	-85
-4	4	122	114
3	4	127*	-68
-3	4	83	-65
2	4	-22	13
-2	4	53	-61
1	4	287	258
-1	4	205	-175
0	4	43	43
8	3	74	-50
-8	3	50	51
7	3	128	-130
-7	3	-42	-7
6	3	165	166
-6	3	38	-24
5	3	-39	37
-5	3	53	52
4	3	81	-74
-4	3	243	193
3	3	21	7
-3	3	114	110
2	3	156	-144
-2	3	129	40
1	3	72	52
-1	3	187	-148
0	3	129	109
-9	2	-30	-33
8	2	-40	19
-8	2	151	-155
7	2	-42	10
-7	2	117	-121
6	2	-40	13
-6	2	156	140
5	2	78	75
-5	2	84	-70
4	2	58	7
-4	2	182	-201
3	2	326*	-251
-3	2	45	-56
2	2	272	-207
-2	2	240	-198
1	2	201	179
-1	2	100	-138
0	2	747	854
9	1	35	33
-9	1	127	-115
8	1	-41	-8
-8	1	267	265
7	1	83	54
-7	1	-42	-19
6	1	52	-49
-6	1	48	29
5	1	173	-164
-5	1	102	90
4	1	-57	14
-4	1	196	222
3	1	121*	-24
-3	1	301	-297
2	1	869	776
-2	1	860	966
1	1	117	81
-1	1	96	71
0	1	110	-57
11	0	117	-114
10	0	180	-163
9	0	102	96
8	0	91	-59
7	0	60	-53
6	0	103	-90
5	0	299	254
4	0	-32	17
3	0	72	51
2	0	417	-539
1	0	185	195
-1	0	167	195
K = 0			
0	14	55	-53
-4	13	55	61
2	13	39	26
-2	13	70	-73
0	13	53	-48
-6	12	39	26
-4	12	53	-57
2	12	64	-53
-2	12	-41	-5
0	12	40	32
8	11	55	52
6	11	41	-44
-6	11	63	48
4	11	43	-38
-4	11	-41	42
2	11	89	94
-2	11	-41	-10
0	11	67	55
8	10	45	53
-8	10	41	46
6	10	96	-87
-6	10	69	67
4	10	78	83
-4	10	-40	5
2	10	77	86
-2	10	102	111
0	10	68	-41
12	9	54	-53
10	9	45	-40
8	9	-44	12
-8	9	41	7
6	9	-42	11
-6	9	-40	-27
4	9	128	-154
-4	9	100	-109
2	9	148	-159
-2	9	79	-96
0	9	-25	24
12	8	79	105
10	8	-41	-43
8	8	57	50
-8	8	41	37
6	8	57	65
-6	8	100	109
4	8	140	-160
-4	8	85	-98
2	8	35	37
-2	8	40	-41
0	8	-23	5
12	7	54	56
10	7	45	51
8	7	-42	-9
-8	7	112	161
6	7	-39	30
-6	7	103	-121
4	7	191	185
-4	7	67*	74
2	7	158	-168
-2	7	108	122
0	7	151	-148
12	6	41	-29
10	6	75	-76
8	6	44	-47
-8	6	124	144
6	6	90	90
-6	6	136	-119
4	6	194	-192
-4	6	112	-109
2	6	34	-36
-2	6	137	109
0	6	193	-145
12	5	55	43
10	5	-42	-4
8	5	121	102
-8	5	194	-208
6	5	-34	-12
-6	5	59*	-31
4	5	137	116
-4	5	77	-80
2	5	380	358
-2	5	-23	-32
0	5	24*	-0
12	4	-62	4
10	4	50	51
8	4	146	-142
-8	4	113	-111
6	4	176	188
-6	4	406	-472
4	4	129	112
-4	4	409	370
2	4	48	-46
-2	4	137	-132
0	4	249	-207
12	3	-43	-23
-12	3	65	71
10	3	-41	29
-10	3	94	120
8	3	76	-72
-8	3	84	-70
6	3	62	65
-6	3	209	185
4	3	73	-49
-4	3	140	124
2	3	234	-224
-2	3	233	-205
0	3	205	164
12	2	45	46
-12	2	101	123
10	2	176	209
-10	2	177	-242
8	2	70	57
-8	2	34	-28
6	2	162	-138
-6	2	49	45
4	2	112	85
-4	2	77	70
2	2	645	613
-2	2	814	711
0	2	403	401
12	1	-43	19
-12	1	60	-68
10	1	92	-109
-10	1	105	-116
8	1	149	150
-8	1	75	-96
6	1	-23	-14
-6	1	35	-29
4	1	-22	29
-4	1	234	-227
2	1	607	741
-2	1	-14	37
0	1	-10	-21
12	0	218	-502
10	0	92*	96
8	0	-32	-20
6	0	336	304
4	0	116	105
2	0	142	180
2	0	142	-180

[a] Reflections designated by an asterisk were assigned zero weight in the refinements when the values to be averaged for a given reflection differed by more than about 20%. A minus sign before F_o indicates a "less than" observation. [b] Divide by 6.5 to place on an absolute scale with $F_{000} = 528$.

and $\langle E_{hkl}^2 \rangle$ (Table II) is normalized to 1.00 by adjustment of the scale factor. Second, the E_{hkl}, abbreviated as E_h, are ordered in decreasing size of $|E_h|\Sigma_{h'}|E_{h'}||E_{h-h'}|$, and the three largest which are linearly independent are fixed in sign in order to determine the origin. Third, the next four reflections are given assumed signs, and each of the 2^N sets ($N = 4$) of assumed signs is then iterated by Sayre's equation through those reflections for which E_{hkl} is greater than some chosen value. In our first attempt, the minimum E_{hkl} was taken as 1.5

Table II

Experimental and Theoretical[a] Values of Normalized Structure Factors

	Exptl	Centrosym	Noncentrosym
$\langle \lvert E \rvert \rangle$	0.841	0.798	0.886
$\langle E^2 - 1 \rangle$	0.897	0.968	0.736
$\lvert E \rvert \geqslant 3.0$, %	0.77	0.3	
$\lvert E \rvert \geqslant 2.0$, %	3.79	5.0	
$\lvert E \rvert \geqslant 1.0$, %	28.38	32.0	

[a] For structures with randomly distributed atoms.

Table III

Disagreement Factors[a] Based on F

Sin θ	No. of reflections	R_F	Class	*hkl* R_F
0.00–0.40	249	0.153	All	0.150
0.40–0.50	207	0.112	$H = 2N$	0.147
0.50–0.60	256	0.125	$H = 2N + 1$	0.153
0.60–0.65	138	0.161	$K = 2N$	0.156
0.65–0.70	141	0.161	$K = 2N + 1$	0.145
0.70–0.75	99	0.154	$L = 2N$	0.155
0.75–0.80	92	0.184	$L = 2N + 1$	0.147
0.80–0.85	91	0.192	$K + L = 2N$	0.149
0.85–0.90	67	0.182	$K + L = 2N + 1$	0.152
0.90–1.00	28	0.206	$L + H = 2N$	0.152
			$L + H = 2N + 1$	0.149
			$H + K = 2N$	0.148
			$H + K = 2N + 1$	0.153
			$H + K + L = 2N$	0.155
			$H + K + L = 2N + 1$	0.146

[a] $R_F = \Sigma \lvert \lvert F_o \rvert - \lvert F_c \rvert \rvert / \Sigma \lvert F_o \rvert = 0.146$; $R_{wF} = [\Sigma w \lvert F_o^2 - F_c^2 \rvert^2 / \Sigma w F_o^4]^{1/2} = 0.165$, calculated excluding methyl H atoms. Including methyl H atoms, $R_F = 0.138$; $R_{wF} = 0.090$; and goodness of fit $= \Sigma w[\lvert F_o \rvert^2 - \lvert F_c \rvert^2]^2/(m - s) = 0.042$, where m is the number of observations and s the number of parameters being refined.

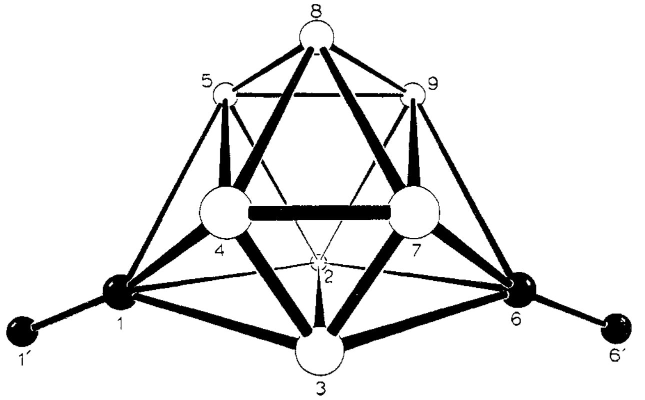

Figure 1.—The molecular structure and numbering scheme for $B_7H_7C_2(CH_3)_2$. Hydrogens are not shown for the sake of clarity. Atoms are designated as: B (2, 3, 4, 5, 6, 7, 8, 9), cage C (1, 6), methyl C (1′, 6′).

(193 reflections), but no set of signs gave a refinable structure. We then lowered the minimum E_{hkl} to 1.2 (326 reflections). Fourth, after each iterative process produces no more sign changes, the sign set with the highest consistency index

$$C = \frac{\langle \lvert E_H \sum_K E_K E_{H-K} \rvert \rangle}{\langle \lvert E_H \rvert \sum_K \lvert E_K \rvert \lvert E_{H-K} \rvert \rangle}$$

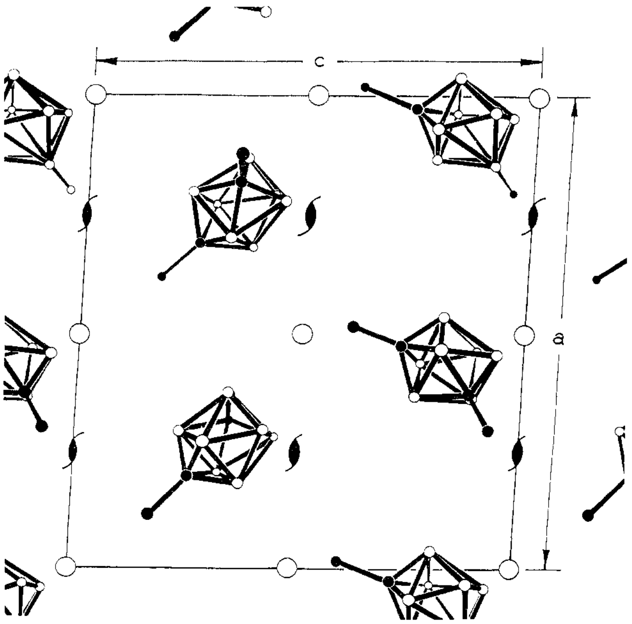

Figure 2.—The molecular packing of $B_7H_7C_2(CH_3)_2$ as seen in the *h0l* projection for the four molecules in the lower half of the unit cell. Hydrogens are omitted for the sake of clarity. Carbons are represented as filled circles.

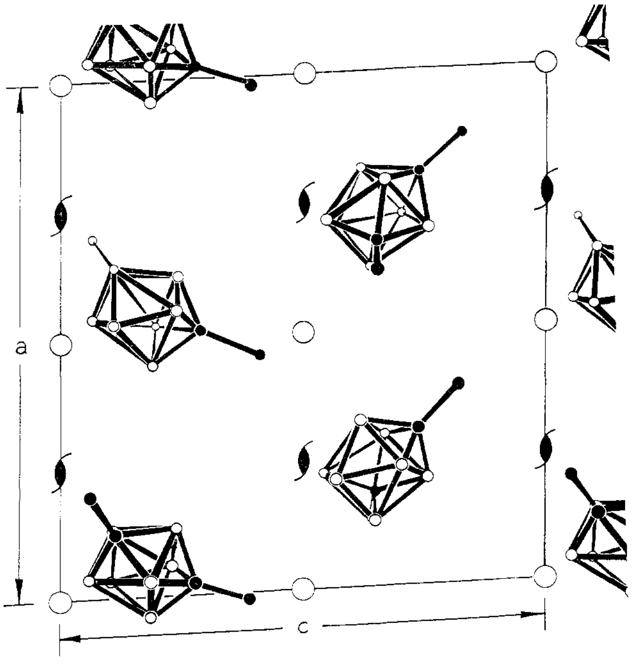

Figure 3.—The molecular packing of $B_7H_7C_2(CH_3)_2$ as in Figure 2 for the four molecules in the upper half of the unit cell.

where the average is taken over all H, is most probably correct. In the iterative process,[10] the newly determined signs were used immediately in the determination of other signs which were further down the list of reflections, but first the program attempted to determine the signs of reflections above on the list which

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(10) In an alternative procedure the newly determined signs were not used to determine signs until the next cycle. After the end of the first cycle for which there were no new additions to the list of signs, the program cycled through the list until no sign changes occurred. This alternative procedure yielded C values which were uniformly lower than those for the procedure described in the text.

TABLE IV

COORDINATES

Atom	x	y	z
C-1_1	0.3072 (5)	0.1923 (6)	0.2561 (6)
B-2_1	0.2029 (7)	0.2547 (7)	0.2334 (7)
B-3_1	0.2954 (6)	0.3121 (7)	0.3257 (7)
B-4_1	0.3215 (6)	0.1474 (7)	0.3789 (7)
B-5_1	0.2270 (6)	0.0919 (7)	0.2871 (8)
C-6_1	0.1803 (9)	0.3263 (8)	0.3426 (8)
B-7_1	0.2206 (7)	0.2506 (7)	0.4474 (7)
B-8_1	0.2102 (7)	0.0997 (7)	0.4215 (8)
B-9_1	0.1271 (7)	0.1953 (7)	0.3542 (7)
C-$1_1'$	0.3830 (6)	0.1772 (7)	0.1726 (7)
C-$6_1'$	0.1243 (6)	0.4446 (7)	0.3469 (8)
H-2_1	0.182 (5)	0.291 (6)	0.156 (5)
H-3_1	0.355 (6)	0.397 (7)	0.321 (6)
H-4_1	0.402 (7)	0.105 (7)	0.404 (7)
H-5_1	0.229 (6)	0.004 (7)	0.242 (6)
H-7_1	0.219 (5)	0.283 (6)	0.530 (5)
H-8_1	0.188 (5)	0.026 (5)	0.434 (5)
H-9_1	0.034 (6)	0.179 (6)	0.367 (6)
CH_3-H-$1_1'$a	0.436 (8)	0.220 (6)	0.199 (5)
CH_3-H-$1_1'$b	0.316 (7)	0.141 (5)	0.093 (7)
CH_3-H-$1_1'$c	0.426 (8)	0.077 (7)	0.177 (6)
CH_3-H-$6_1'$a	0.139 (10)	0.510 (6)	0.357 (6)
CH_3-H-$6_1'$b	0.086 (9)	0.481 (7)	0.279 (5)
CH_3-H-$6_1'$c	0.068 (8)	0.462 (5)	0.405 (6)
C-1_2	0.5241 (6)	0.3097 (6)	0.7232 (6)
B-2_2	0.6341 (6)	0.3337 (6)	0.7644 (6)
B-3_2	0.5343 (7)	0.4121 (7)	0.8141 (7)
B-4_2	0.4592 (9)	0.2586 (9)	0.8177 (9)
B-5_2	0.5629 (8)	0.1829 (7)	0.7706 (8)
C-6_2	0.6301 (6)	0.3780 (6)	0.8852 (5)
B-7_2	0.5428 (9)	0.3129 (8)	0.9437 (8)
B-8_2	0.5331 (10)	0.1714 (8)	0.8993 (10)
B-9_2	0.6432 (7)	0.2346 (7)	0.8957 (7)
C-$1_2'$	0.4825 (7)	0.3334 (9)	0.6069 (7)
C-$6_2'$	0.7011 (9)	0.4700 (6)	0.9389 (7)
H-2_2	0.689 (5)	0.363 (9)	0.703 (5)
H-3_2	0.503 (5)	0.509 (6)	0.810 (5)
H-4_2	0.388 (9)	0.241 (10)	0.818 (8)
H-5_2	0.575 (6)	0.112 (7)	0.733 (6)
H-7_2	0.526 (8)	0.359 (10)	1.021 (9)
H-8_2	0.502 (6)	0.906 (7)	0.926 (6)
H-9_2	0.719 (7)	0.204 (7)	0.937 (7)
CH_3-H-$1_2'$a	0.444 (8)	0.404 (7)	0.613 (6)
CH_3-H-$1_2'$b	0.399 (8)	0.295 (6)	0.632 (5)
CH_3-H-$1_2'$c	0.553 (7)	0.357 (5)	0.549 (5)
CH_3-H-$6_2'$a	0.758 (9)	0.432 (6)	0.938 (7)
CH_3-H-$6_2'$b	0.693 (9)	0.469 (6)	1.038 (6)
CH_3-H-$6_2'$c	0.629 (8)	0.542 (6)	0.925 (5)

were still undetermined. The set of signs which yielded the correct structure had the highest consistency index $C = 0.63$ which was reached in three cycles. The next best set had a consistency index of 0.62 and required six cycles of iteration to reach self-consistency. Not only did the starting sign set remain invariant, but our final refinements, described below, changed only one of these 326 signs.

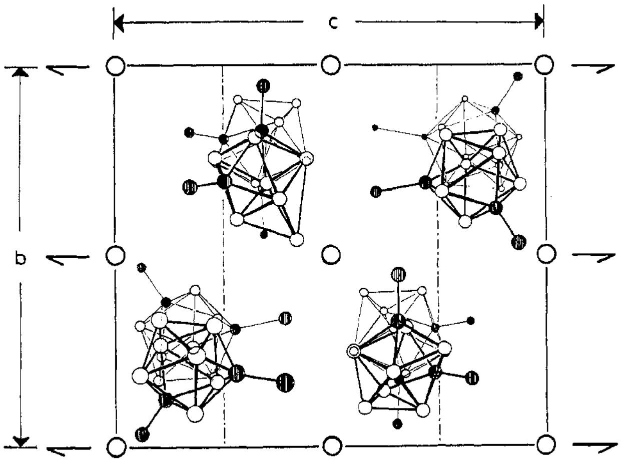

Figure 4.—Molecular packing of entire unit cell shown in the $0kl$ projection. Carbons are represented as hatched circles. The upper molecules are drawn in heavy lines; the lower in light lines.

TABLE V

THERMAL PARAMETERS, ANISOTROPIC[a] ($\times 10^4$) AND ISOTROPIC[b]

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C-1_1	16	36	58	9	−19	−15
B-2_1	36	42	55	−22	16	−10
B-3_1	12	56	44	−15	26	12
B-4_1	27	29	63	−5	−13	1
B-5_1	24	43	70	−2	14	−10
C-6_1	14	32	49	8	3	−9
B-7_1	24	44	50	−8	14	5
B-8_1	30	22	73	0	−13	38
B-9_1	39	40	52	−30	−10	1
C-$1_1'$	36	94	57	1	54	−32
C-$6_1'$	26	44	101	35	−15	−10
C-1_2	33	49	57	−5	14	−28
B-2_2	11	32	47	−3	3	1
B-3_2	30	45	49	21	10	19
B-4_2	61	76	96	−91	70	−53
B-5_2	43	29	89	−1	66	−31
C-6_2	35	44	39	−0	3	2
B-7_2	71	40	64	15	6	10
B-8_2	94	33	111	8	96	59
B-9_2	47	35	56	53	26	32
C-$1_2'$	54	143	59	27	−41	−48
C-$6_2'$	60	81	93	−25	−52	−39

Atom	B	Atom	B
H-2_1	0.11	H-2_2	0.29
H-3_1	2.31	H-3_2	1.57
H-4_1	2.67	H-4_2	6.38
H-5_1	2.42	H-5_2	2.56
H-7_1	0.76	H-7_2	6.32
H-8_1	0.11	H-8_2	2.14
H-9_1	2.05	H-9_2	3.50
CH_3-H-$1_1'$a	12.24	CH_3-H-$1_2'$a	16.41
CH_3-H-$1_1'$b	9.46	CH_3-H-$1_2'$b	3.85
CH_3-H-$1_1'$c	3.14	CH_3-H-$1_2'$c	12.90
CH_3-H-$6_1'$a	1.00	CH_3-H-$6_2'$a	7.84
CH_3-H-$6_1'$b	12.77	CH_3-H-$6_2'$b	8.67
CH_3-H-$6_1'$c	9.42	CH_3-H-$6_2'$c	−1.40

[a] Anisotropic temperature factors are calculated by the expression $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + kl\beta_{23})]$.
[b] Isotropic temperature factors are given by $\exp[-B(\sin^2\theta)/\lambda^2]$.

A Fourier map computed from these 326 E values and signs yielded 22 peaks in the asymmetric unit and was readily interpretable as two independent B_7C_4 molecules. Heights ranged from 23 to 40, on an arbitrary scale on which the next highest peak was 16. These atoms gave a value of $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.32$ for all 1883 reflections before refinement, where only those below-minimum F_o's for which $F_c > F_o$ were included. Position and isotropic thermal parameters on B and C were refined through three cycles of least-squares minimization of $\Sigma w^2(k^2|F_o|^2 - |F_c|^2)^2$, where $w = 1/F_o^2$ to yield $R = 0.27$. Terminal H atoms on B

Table VI

Maximum Value of σ for Temperature Factors (Anisotropic, $\times 10^4$)

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	B
Apical B	10	8	10	14	18	13	
Equatorial B	8	10	10	13	15	14	
B bonded to two C's	6	8	7	9	10	9	
Carborane C	5	7	5	8	9	8	
Methyl C	7	12	8	13	12	12	
Cage H							2.92
Methyl H							2.16

Table VII

Complete Bond Distances

Bond	Bond length, Å Molecule 1	 Molecule 2
	Apical B–B	
B(8)–B(4)	1.72 (1)	1.69 (2)
B(8)–B(5)	1.71 (1)	1.68 (2)
B(8)–B(7)	1.71 (1)	1.67 (1)
B(8)–B(9)	1.73 (1)	1.66 (2)
	B–B between B's Connected to Both C's	
B(2)–B(3)	1.77 (1)	1.75 (1)
	B–B at Base of Apical B Cap, 1st Type	
B(4)–B(5)	1.78 (1)	1.77 (1)
B(7)–B(9)	1.78 (1)	1.75 (1)
	B–B at Base of Apical B Cap, 2nd Type	
B(4)–B(7)	2.01 (1)	1.98 (2)
B(5)–B(9)	2.00 (1)	1.94 (1)
	B–B at Base of C Caps	
B(2)–B(5)	1.95 (1)	1.94 (1)
B(2)–B(9)	1.98 (1)	1.97 (1)
B(3)–B(4)	1.97 (1)	1.99 (1)
B(3)–B(7)	1.99 (1)	1.95 (1)
	B–C B's Bonded to One C	
C(1)–B(4)	1.61 (1)	1.61 (1)
C(1)–B(5)	1.62 (1)	1.60 (1)
C(6)–B(7)	1.62 (1)	1.60 (1)
C(6)–B(9)	1.63 (1)	1.61 (1)
	B–C B's Bonded to Two C's	
C(1)–B(2)	1.59 (1)	1.58 (1)
C(1)–B(3)	1.60 (1)	1.61 (1)
C(6)–B(2)	1.62 (1)	1.59 (1)
C(6)–B(3)	1.60 (1)	1.58 (1)
	C–C Cage C to Methyl C	
C(1)–C(1′)	1.51 (1)	1.55 (1)
C(6)–C(6′)	1.52 (1)	1.53 (1)
	B–H Apical	
B(8)–H	1.18 (6)	1.00 (8)
	B–H B Bonded to One C	
B(4)–H	1.22 (9)	0.99 (12)
B(5)–H	1.13 (8)	0.94 (8)
B(7)–H	1.09 (7)	1.12 (11)
B(9)–H	1.30 (8)	1.17 (9)
	B–H B Bonded to Two C's	
B(2)–H	1.07 (6)	1.14 (6)
B(3)–H	1.25 (8)	1.16 (7)

Table VIII

Bond Summary[a]

Bond		Symmetry-related bonds[b]	Mean value, Å	Std dev calcd from coordinate dev, Å Av[c] dev	 Max dev	Std dev of the mean,[d] Å
4–7	B–B	5–9	1.984	0.013	0.015	0.031
4–8	B–B	5–8, 7–8, 9–8	1.698	0.014	0.016	0.026
3–2	B–B		1.764	0.012	0.012	0.049
4–5	B–B	7–9	1.772	0.013	0.015	0.046
2–5	B–B	2–9, 3–4, 3–7	1.969	0.012	0.013	0.021
1–4	C–B	1–5, 6–7, 6–9	1.615	0.011	0.013	0.036
1–2	C–B	1–3, 6–2, 6–3	1.595	0.010	0.011	0.015
1–1′	C–C	6–6′	1.529	0.011	0.011	0.015
B–H, apical B			1.093	0.067	0.075	0.090
B–H, B bonded to one C			1.119	0.091	0.123	0.120
B–H, B bonded to both C's			1.154	0.069	0.082	

[a] For the boron–carbon framework: average standard deviation calculated from atomic coordinates, 0.012 Å; average standard deviation of the mean for all symmetry types, 0.029 Å. [b] Assuming C_{2v} symmetry, we have averaged distances for equivalent bonds, including both molecules. [c] Average of deviations for equivalent bonds. [d] Calculated by the formula

$$\sigma = \frac{\left(\sum_{i=1}^{N}(D_i - \bar{D})^2\right)^{1/2}}{N - 1}$$

where D_i are the individual bond distances, $\bar{D}$ is the mean distance, and N is the number of equivalent bonds.

atoms were introduced at a B–H distance of 1.1 Å on a model, and then anisotropic thermal parameters for B and C and isotropic thermal parameters for H were refined alternately to $R = 0.146$. A difference map from which B, C, and H (on B) were subtracted yielded density resembling methyl H atoms at about half the height of H atoms attached to B. Introduction of these peaks and subsequent least-squares refinements gave $R = 0.138$ (Table III), but the thermal parameters of methyl hydrogen atoms showed considerable variation and values suggestive of disorder. A final difference electron density map showed no uninterpretable peaks higher than 0.20 electron/Å^3.

Geometrical Structure

The molecule (Figure 1), properly numbered,[11] has H atoms extending approximately away from the molecular center on each B atom. For clarity, we show the four molecules in the lower half of the unit cell in Figure 2, and those in the upper half in Figure 3. A different projection of the whole unit cell is shown in Figure 4. The parameters of Tables IV–VI yield the bond distances and angles of Tables VII–X. We have calculated intermolecular distances between all pairs of atoms in one unit cell. The closest approaches are 2.30 Å (methyl H–cage H) and 2.60 Å (cage H–cage H). In view of the large uncertainty in methyl H positions, the packing seems reasonable.

The molecular structure of $B_7C_2H_7(CH_3)_2$ is a tricapped trigonal prism, as suggested earlier,[1] with the two C atoms and their attached methyl groups located at two of the apices. There are three types of B atoms: a five-coordinated apical B, four six-coordinated equatorial B atoms bonded to one C each, and two six-coordinated B atoms each bonded to two C atoms, all in agreement with the ^{11}B nuclear magnetic resonance results.[1] Since there are two molecules per asymmetric unit and no molecular symmetry imposed by the space group,

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(11) R. Adams, *Inorg. Chem.*, **2**, 1087 (1963).

TABLE IX

COMPLETE BOND ANGLES

Angle	Molecule 1	Molecule 2
Among Three B's All Bound to Apical B		
B(9)–B(7)–B(4)	90.2 (5)	90.1 (6)
B(9)–B(5)–B(4)	90.8 (5)	90.9 (6)
B(7)–B(9)–B(5)	89.7 (6)	90.5 (6)
B(7)–B(4)–B(5)	89.2 (5)	88.6 (7)
Among Three B's with Central B Connected to Apical C and B		
B(9)–B(7)–B(3)	89.9 (5)	90.5 (6)
B(7)–B(9)–B(2)	89.9 (6)	89.5 (6)
B(5)–B(4)–B(3)	89.8 (5)	88.9 (6)
B(4)–B(5)–B(2)	90.0 (5)	90.6 (5)
Among Three B's with Central B Connected to Two C's		
B(7)–B(3)–B(2)	89.8 (5)	90.1 (5)
B(9)–B(2)–B(3)	90.4 (5)	89.9 (5)
B(5)–B(2)–B(3)	90.6 (5)	91.0 (5)
B(4)–B(3)–B(2)	89.6 (5)	89.5 (5)
Around Apical B, 1st Type		
B(7)–B(8)–B(4)	72.0 (5)	72.2 (6)
B(9)–B(8)–B(5)	71.0 (5)	71.1 (7)
Around Apical B, 2nd Type		
B(7)–B(8)–B(9)	62.3 (5)	63.5 (7)
B(4)–B(8)–B(5)	62.8 (5)	63.2 (6)
Around Cage C, 1st Type		
B(3)–C(6)–B(2)	66.9 (5)	67.2 (5)
B(3)–C(1)–B(2)	67.6 (5)	66.8 (5)
Around Cage C, 2nd Type		
B(7)–C(6)–B(9)	66.3 (5)	66.2 (6)
B(4)–C(1)–B(5)	66.9 (5)	66.7 (6)
Around Cage C, 3rd Type		
B(7)–C(6)–B(3)	76.5 (5)	75.9 (6)
B(9)–C(6)–B(2)	75.1 (5)	76.2 (5)
B(4)–C(1)–B(3)	75.7 (5)	76.3 (6)
B(5)–C(1)–B(2)	74.9 (5)	75.1 (6)
Angles in the Trigonal Prism, 1st Type		
B(3)–B(7)–B(4)	58.9 (4)	60.7 (5)
B(2)–B(9)–B(5)	58.7 (4)	59.4 (4)
B(3)–B(4)–B(7)	60.0 (4)	59.0 (5)
B(2)–B(5)–B(9)	60.3 (4)	61.1 (5)
Angles in the Trigonal Prism, 2nd Type		
B(4)–B(3)–B(7)	61.0 (4)	60.3 (5)
B(5)–B(2)–B(9)	61.0 (4)	59.5 (5)
Angles Including Methyl C's, 1st Type		
C(6′)–C(6)–B(7)	124.5 (6)	124.5 (7)
C(6′)–C(6)–B(9)	122.8 (6)	124.1 (7)
C(1′)–C(1)–B(5)	125.4 (6)	126.1 (7)
C(1′)–C(1)–B(4)	124.6 (6)	123.9 (8)
Angles Including Methyl C's 2nd Type		
C(6′)–C(6)–B(3)	125.8 (6)	124.4 (6)
C(6′)–C(6)–B(2)	124.9 (6)	124.5 (7)
C(1′)–C(1)–B(3)	123.8 (6)	123.5 (7)
C(1′)–C(1)–B(2)	124.1 (7)	124.5 (7)
Angles at Base of Apical B Cap, 1st Type		
B(8)–B(7)–B(4)	54.1 (5)	54.5 (6)
B(8)–B(5)–B(9)	55.1 (5)	53.8 (6)
B(8)–B(9)–B(5)	53.9 (5)	55.1 (6)
B(8)–B(4)–B(7)	53.9 (4)	53.3 (6)

Angle	Molecule 1	Molecule 2
Angles at Base of Apical B Cap, 2nd Type		
B(8)–B(7)–B(9)	59.4 (5)	57.8 (7)
B(8)–B(9)–B(7)	58.3 (5)	58.6 (6)
B(8)–B(5)–B(4)	58.8 (5)	58.7 (7)
B(8)–B(4)–B(5)	58.4 (5)	58.1 (7)
Angles at Base of C Cap, 1st Type		
B(3)–B(7)–C(6)	51.2 (4)	51.6 (5)
B(2)–B(9)–C(6)	52.1 (4)	51.5 (4)
C(1)–B(4)–B(3)	51.9 (4)	51.7 (5)
C(1)–B(5)–B(2)	51.8 (4)	51.8 (4)
Angles at Base of C Cap, 2nd Type		
C(6)–B(7)–B(9)	57.2 (4)	57.1 (5)
C(6)–B(9)–B(7)	56.5 (5)	56.7 (5)
C(1)–B(5)–B(4)	56.3 (5)	56.9 (5)
C(1)–B(4)–B(5)	56.8 (5)	56.4 (5)
Angles at Base of C Cap, 3rd Type		
C(6)–B(3)–B(7)	52.3 (4)	52.5 (5)
C(6)–B(2)–B(9)	52.8 (4)	52.4 (4)
C(1)–B(3)–B(4)	52.4 (4)	52.0 (5)
C(1)–B(2)–B(5)	53.3 (4)	53.1 (5)
Angles at Base of C Cap, 4th Type		
C(6)–B(3)–B(2)	57.2 (5)	56.6 (5)
C(6)–B(2)–B(3)	55.9 (5)	56.2 (5)
C(1)–B(3)–B(2)	56.6 (5)	55.8 (5)
C(1)–B(2)–B(3)	55.9 (5)	57.4 (5)
Diagonally across Apical B Cap		
B(9)–B(8)–B(4)	77.1 (6)	75.7 (7)
B(7)–B(8)–B(5)	77.2 (6)	76.9 (7)
Diagonally across Apical C Cap		
B(7)–C(6)–B(2)	69.5 (6)	69.0 (6)
B(9)–C(6)–B(3)	68.6 (6)	68.4 (6)
B(4)–C(1)–B(2)	68.7 (6)	68.4 (7)
B(5)–C(1)–B(3)	69.3 (6)	69.6 (6)
Angles Connecting Cage C and Apical B		
C(1)–B(4)–B(8)	109.2 (6)	108.1 (9)
C(1)–B(5)–B(8)	109.1 (6)	109.0 (7)
C(6)–B(7)–B(8)	109.6 (6)	108.7 (8)
C(6)–B(9)–B(8)	108.0 (7)	109.0 (7)
Angles Connecting Two Cage C's		
C(1)–B(3)–C(6)	106.3 (6)	105.2 (6)
C(1)–B(2)–C(6)	105.9 (6)	106.3 (6)
Angles Connecting Cage C and Nonapical B, 1st Type		
C(6)–B(7)–B(4)	99.3 (5)	100.5 (6)
C(6)–B(9)–B(5)	99.3 (6)	99.5 (6)
C(1)–B(4)–B(7)	100.2 (5)	99.2 (7)
C(1)–B(5)–B(9)	100.7 (5)	101.2 (6)
Angles Connecting Cage C and Nonapical B, 2nd Type		
C(6)–B(3)–B(4)	101.9 (5)	100.9 (6)
C(6)–B(2)–B(5)	101.7 (6)	100.8 (6)
C(1)–B(3)–B(7)	101.4 (5)	100.6 (5)
C(1)–B(2)–B(9)	102.4 (6)	101.0 (6)
Angles Connecting Apical B and Nonapical B		
B(8)–B(5)–B(2)	105.1 (6)	104.5 (6)
B(8)–B(9)–B(2)	102.7 (6)	104.1 (7)
B(8)–B(7)–B(3)	103.4 (6)	104.9 (7)
B(8)–B(4)–B(3)	104.3 (6)	102.5 (7)

TABLE X

ANGLE SUMMARY[a]

Angle	Symmetry-related angles	Mean value, deg	Dev calcd from coordinate dev, deg: Av dev	Dev calcd from coordinate dev, deg: Max dev	Std dev of the mean, deg
9–7–4	9–5–4, 7–9–5, 7–4–5	90.00	0.59	0.68	0.65
9–7–3	7–9–2, 5–4–3, 4–5–2	89.89	0.55	0.62	0.38
7–3–2	9–2–3, 5–2–3, 4–3–2	90.11	0.53	0.55	0.54
7–8–4	9–8–5	71.58	0.60	0.70	0.59
7–8–9	4–8–5	62.97	0.57	0.66	0.52
3–6–2	3–1–2	67.13	0.51	0.52	0.24
7–6–9	4–1–5	66.54	0.54	0.60	0.22
7–6–3	9–6–2, 4–1–3, 5–1–2	75.69	0.54	0.61	0.61
3–7–4	2–9–5, 3–4–7, 2–5–9	59.77	0.44	0.49	0.89
4–3–7	5–2–9	60.47	0.45	0.50	0.56
6′–6–7	6′–6–9, 1′–1–5, 1′–1–4	124.51	0.66	0.75	0.99
6′–6–3	6′–6–2, 1′–1–3, 1′–1–2	124.44	0.63	0.68	0.71
8–7–4	8–5–9, 8–9–5, 8–4–7	54.21	0.52	0.61	0.63
8–7–9	8–9–7, 8–5–4, 8–4–5	58.51	0.55	0.65	0.50
3–7–6	2–9–6, 1–4–3, 1–5–2	51.71	0.43	0.47	0.28
6–7–9	6–9–7, 1–5–4, 1–4–5	56.73	0.48	0.54	0.29
6–3–7	6–2–9, 1–3–4, 1–2–5	52.60	0.44	0.48	0.45
6–3–2	6–2–3, 1–3–2, 1–2–3	56.44	0.46	0.48	0.61

[a] Average of all standard deviations calculated from atomic coordinates, 0.53°; average of all standard deviations of the mean for all symmetry types, 0.54°; average maximum deviation calculated from atomic coordinates for each symmetry type, 0.58°.

we obtained two independent determinations of all bond distances and angles (Tables VII–X). Comparison of these molecules indicates that bond distances are known to within ±0.03 Å and angles to within ±0.6°.

It is interesting to note that the introduction of two C atoms into the cage has not greatly distorted the remaining B–B bonds from the full D_{3h} symmetry reported[12] for the isoelectronic species $B_9H_9^{2-}$. Bond B_4–B_7 is equal to B_2–B_5, and bond B_2–B_3 is equal to B_4–B_5 to within a standard deviation. In accordance with previous observations,[13,14] we found the shortest B–B distances to be those involving the apical B (B_4–B_8, etc.). However, we do not observe abnormal lengthening of the B_2–B_3 distance between B atoms each joined to two C atoms. This distance is 1.76 Å, as compared to 1.89 ± 0.01 Å in the related compound, $B_6H_6C_2(CH_3)_2$,[14] 1.89 ± 0.04 Å in *m*-$B_{10}Br_2H_8C_2H_2$,[15] and 1.89 ± 0.06 Å in *m*-$B_{10}Cl_{10}C_2H_2$[16] for the same type of distance.

Molecular Orbital Studies

Several molecular orbital (MO) studies were carried out for $B_7H_7C_2(CH_3)_2$. The molecules as determined by X-ray diffraction correspond to C_{2v} symmetry within the standard deviations of bond lengths. Therefore, no attempt was made to idealize the molecule to C_{2v} symmetry. However, the uncertain H atom positions were adjusted to yield the following bond lengths: C–H, 1.095 Å; apical B–H, 1.210 Å; and equatorial B–H, 1.250 Å. The H positions of CH_3 groups were chosen to give a tetrahedral arrangement of bonds around the C. The complete set of adjusted orthogonalized atomic coordinates is given in Table XI.

TABLE XI

ORTHOGONALIZED COORDINATES[a]

Molecule 1: No.	*x*	*y*	*z*	Molecule 2: No.	*x*	*y*	*z*
B(8)	2.5640	1.1074	5.2471	B(8)	6.6209	1.9042	11.1954
B(4)	4.1061	1.6375	4.7170	B(4)	5.6735	2.8734	10.1792
B(5)	2.8864	1.0203	3.5744	B(5)	7.1153	2.0315	9.5925
B(7)	2.6869	2.7841	5.5694	B(7)	6.7221	3.4764	11.7484
B(9)	1.4816	2.1694	4.4089	B(9)	8.1201	2.6062	11.1506
B(2)	2.5961	2.8300	2.9058	B(2)	8.0869	3.7075	9.5162
B(3)	3.7892	3.4673	4.0548	B(3)	6.6969	4.5781	10.1342
C(1)	3.9973	2.1361	3.1885	C(1)	6.6209	3.4401	9.0025
C(6)	2.2119	3.6250	4.2654	C(6)	7.9485	4.1996	11.0196
C(1′)	5.0855	1.9684	2.1487	C(1′)	6.1366	3.7040	7.5557
C(6′)	1.4485	4.9386	4.3185	C(6′)	8.8774	5.2209	11.6887
H(8)	2.2064	0.2654	6.0389	H(8)	6.0843	0.8937	11.5896
H(4)	5.2109	1.1499	5.0405	H(4)	4.4484	2.6249	10.1800
H(5)	2.9460	−0.0581	2.9450	H(5)	7.3608	0.9726	8.9750
H(7)	2.5983	3.1974	6.7459	H(7)	6.4191	4.0492	12.8150
H(9)	0.2535	1.9946	4.5638	H(9)	9.1864	2.2461	11.6947
H(2)	2.3262	3.2980	1.7785	H(2)	8.9443	4.0644	8.6793
H(3)	4.6071	4.4111	4.0008	H(3)	6.2471	5.7434	10.0836
H(1′a)[b]	6.0637	1.9684	2.6409	H(1′a)	5.0419	3.7040	7.5316
H(1′b)	5.0370	2.7956	1.4330	H(1′b)	6.5090	4.6767	7.2178
H(1′c)	4.9427	1.0203	1.6205	H(1′c)	6.5162	2.9179	6.8946
H(6′a)	0.7821	4.9386	5.1875	H(6′a)	9.8468	5.2209	11.1796
H(6′b)	2.1570	5.7694	4.4018	H(6′b)	9.0777	4.9523	12.7409
H(6′c)	0.8564	5.0538	3.4046	H(6′c)	8.4306	6.2184	11.6231

[a] As adjusted to give an idealized model for the purposes of MO calculations. [b] H(1′a), H(1′b), etc., are the calculated CH_3 H positions.

TABLE XII

AVERAGE NET MULLIKEN CHARGE FOR EACH KIND OF ATOM IN THE MOLECULE (C_{2v} SYMMETRY ASSUMED)[a]

Kind (numbers)	I	II	III	IV
Apical B	−0.001	−0.105	−0.205	0.021
Equatorial B (4, 5, 7, 9)	0.355	0.156	−0.040	−0.234
B bonded to two C's (2, 3)	0.623	0.396	0.169	−0.127
Cage C (1, 6)	−0.472	0.288	0.163	1.024
Methyl C (1′, 6′)	−0.163	−0.322	−0.314	−0.378
(B)H apical	−0.226	−0.216	−0.017	−0.117
(B)H equatorial	−0.245	−0.247	−0.047	−0.115
(B)H B bonded to two C's	−0.243	−0.267	−0.048	−0.163
H methyl	0.049	0.082	0.105	0.130

[a] I, Hoffmann's extended Hückel; II, NEMO I, K^{ZO} = 0.229; III, NEMO I, K^{ZO} = 0.000; IV, NEMO II, Boyd.

The molecular orbital methods are the extended Hückel method[17,18] in which the Wolfsberg–Helmholz[19] approximation is used, the NEMO (nonempirical molecular orbital) I method,[20] and the NEMO II[21] method. These NEMO methods are based upon self-consistent field results for B_4H_4[22] and C_2H_6,[23] and the NEMO I method was carried out with the single-center zero-overlap constant[24] set equal to K^{ZO} = 0.229 and

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TABLE XIII

FRAMEWORK CHARGES.[a] AVERAGE NET MULLIKEN CHARGE FOR EACH KIND OF CAGE UNIT (C_{2v} SYMMETRY ASSUMED)[b]

Kind	I	II	III	IV
Apical B–H	−0.277	−0.321	−0.222	−0.096
Equatorial B–H	0.111	−0.091	−0.087	−0.349
B–H bonded to two C's	0.380	0.129	0.120	−0.290
C–CH_3	−0.489	0.213	0.164	1.037

[a] Sum of charges on cage atom and on attached H or CH_3. [b] I, Hoffmann's extended Hückel; II, NEMO I, $K^{ZO} = 0.229$; III, NEMO I, $K^{ZO} = 0.000$; IV, NEMO II, Boyd.

$K^{ZO} = 0$. The methods and parameters are the same as those described in the preceding paper.[14]

The average net Mulliken charges[25] for each type of atom in $B_7H_7C_2(CH_3)_2$ are summarized in Table XII for the four MO calculations. From studies of the more symmetrical $B_8C_2H_{10}$,[26] it has been predicted that the most negative B atoms will be those bonded to no C atoms, followed by B atoms bonded to one C, with B atoms bonded to two C's being least negative. Our charges follow this order, $B_8 < B_4, B_5, B_7, B_9 < B_2, B_3$, in all cases, except for the NEMO II calculation where B_8 becomes most positive, the equatorial B atoms most negative, and B_2 and B_3 assume an intermediate value. This method also predicts a charge of 1.024 for the cage C atoms, perhaps indicating that the method anomalously transfers electrons to B_2 and B_3. As has been previously noted,[27] the major effect of including zero-overlap matrix elements in NEMO I calculations is to increase the number of electrons of the framework H atoms at the expense of cage B atoms. A calculation of framework charges, presented in Table XIII, produces no changes in the relative order of charges on B.

TABLE XIV

CUMULATIVE ORBITAL CHARGES FOR $B_7C_2H_7(CH_3)_2$ FROM VARIOUS MOLECULAR ORBITAL CALCULATIONS[a]

		1	2	3	4	5	6	7	8	9	10
Extended Hückel											
Highest filled MO											
Apical B	(8)	0.681	0.712	1.243	1.303	1.486	1.506	1.512	1.734	1.742	1.784
Equatorial B	(4)	0.244	0.596	0.803	0.878	0.987	1.098	1.239	1.325	1.434	1.520
B bonded to two C's	(2)	0.014	0.329	0.333	0.499	0.627	0.802	0.972	1.126	1.143	1.237
Lowest empty MO											
Apical B	(8)	0.448	0.509	0.837	0.837	0.839	1.068	1.363	1.426	1.427	1.646
Equatorial B	(4)	0.139	0.447	0.617	0.968	1.416	1.549	1.635	1.725	1.854	2.011
B bonded to two C's	(2)	0.197	0.628	0.710	0.986	1.038	1.555	1.672	1.781	2.068	2.174
NEMO $K^{ZO} = 0.000$											
Highest filled MO											
Apical B	(8)	0.341	0.572	0.761	0.980	1.156	1.249	1.277	1.327	1.416	1.510
Equatorial B	(4)	0.116	0.296	0.396	0.447	0.633	0.655	0.893	1.075	1.210	1.241
B bonded to two C's	(2)	0.123	0.208	0.370	0.393	0.456	0.601	0.647	0.942	1.077	1.146
Lowest empty MO											
Apical B	(8)	0.419	0.702	0.797	0.812	0.833	1.042	1.219	1.277	1.408	1.432
Equatorial B	(4)	0.120	0.507	0.509	0.823	1.269	1.343	1.485	1.614	1.811	1.887
B bonded to two C's	(2)	0.128	0.239	0.544	0.789	0.827	1.292	1.356	1.652	1.881	2.021
NEMO $K^{ZO} = 0.229$											
Highest Filled MO											
Apical B	(8)	0.355	0.537	0.708	0.995	1.123	1.183	1.307	1.360	1.368	1.446
Equatorial B	(4)	0.115	0.245	0.376	0.425	0.587	0.700	0.864	0.965	1.103	1.130
B bonded to two C's	(2)	0.115	0.208	0.298	0.359	0.429	0.517	0.829	0.930	0.985	1.067
Lowest empty MO											
Apical B	(8)	0.395	0.670	0.839	0.842	0.871	1.074	1.193	1.198	1.200	1.203
Equatorial B	(4)	0.135	0.558	0.563	0.881	1.276	1.351	1.435	1.498	1.558	1.669
B bonded to two C's	(2)	0.151	0.336	0.644	0.947	1.030	1.435	1.512	1.554	1.714	1.744
NEMO II, Boyd											
Highest filled MO											
Apical B	(8)	0.430	0.786	0.796	0.865	1.092	1.226	1.259	1.263	1.268	1.341
Equatorial B	(4)	0.163	0.468	0.555	0.796	1.047	1.163	1.182	1.191	1.234	1.292
B bonded to two C's	(2)	0.092	0.117	0.404	0.668	0.949	1.135	1.271	1.553	1.567	1.631
Lowest empty MO											
Apical B	(8)	0.501	0.622	0.710	0.726	0.963	1.075	1.421	1.569	1.641	1.682
Equatorial B	(4)	−0.029	0.057	0.291	0.484	0.786	0.987	1.237	1.326	1.431	1.632
B bonded to two C's	(2)	−0.023	0.179	0.258	0.282	0.374	0.518	0.571	0.773	0.913	0.976

[a] Cumulative charges are given in units of electrons assuming two electrons per atomic orbital. These charges do not represent a net charge since no core charge has been subtracted.

In the NEMO calculations there are 68 total orbitals. Orbital 33 is the filled orbital of highest energy (HFMO) while orbital 68 is the most stable. The extended Hückel method does not explicitly consider 1s orbitals for C and B. Therefore, there are 57 total orbitals; orbital 33 again is the highest filled molecular orbital with orbital 57 the most stable. Filled molecular orbitals with the highest energy are expected to be the most polarizable, and therefore might dominate in electro-

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Table XV

Cumulative Framework Charges for $B_7C_2H_7(CH_3)_2$ from Various Molecular Orbital Calculations

	1	2	3	4	5	6	7	8	9	10
Extended Hückel										
Highest filled MO										
Apical B–H	0.681	0.734	1.265	1.333	1.646	1.681	1.688	2.039	2.047	2.117
Equatorial B–H	0.259	0.614	0.851	0.958	1.067	1.202	1.431	1.577	1.750	1.907
B–H bonded to two C's	0.014	0.335	0.348	0.518	0.663	0.846	1.155	1.415	1.433	1.595
Lowest empty MO										
Apical B–H	0.448	0.512	0.840	0.840	0.842	1.072	1.373	1.467	1.468	1.688
Equatorial B–H	0.141	0.449	0.620	0.971	1.419	1.554	1.662	1.753	1.884	2.041
B–H bonded to two C's	0.197	0.628	0.710	0.986	1.038	1.555	1.680	1.841	2.126	2.242
NEMO I, $K^{ZO} = 0.000$										
Highest filled MO										
Apical B–H	0.341	0.689	1.050	1.374	1.561	1.697	1.728	1.796	2.052	2.263
Equatorial B–H	0.126	0.354	0.468	0.678	0.881	0.924	1.366	1.607	1.759	1.822
B–H bonded to two C's	0.123	0.213	0.376	0.425	0.488	0.771	0.826	1.524	1.659	1.795
Lowest empty MO										
Apical B–H	0.419	0.728	0.843	0.859	0.883	1.109	1.538	1.664	1.916	1.944
Equatorial B–H	0.133	0.538	0.544	0.860	1.286	1.362	1.653	1.933	2.287	2.368
B–H bonded to two C's	0.134	0.250	0.569	0.821	0.859	1.324	1.409	1.978	2.396	2.667
NEMO I, $K^{ZO} = 0.229$										
Highest filled MO										
Apical B–H	0.355	0.606	0.803	1.452	1.620	1.684	1.992	2.154	2.163	2.376
Equatorial B–H	0.125	0.314	0.605	0.740	0.912	1.182	1.391	1.496	1.773	1.828
B–H bonded to two C's	0.115	0.218	0.329	0.444	0.515	0.604	1.452	1.651	1.781	1.891
Lowest empty MO										
B–H apical	0.395	0.687	0.866	0.870	0.899	1.106	1.385	1.390	1.394	1.398
Equatorial B–H	0.145	0.570	0.579	0.897	1.295	1.374	1.485	1.548	1.618	1.729
B–H bonded to two C's	0.155	0.340	0.648	0.951	1.035	1.444	1.535	1.580	1.751	1.789
NEMO II, Boyd										
Highest filled MO										
Apical B–H	0.430	0.787	0.799	0.880	1.122	1.320	1.354	1.364	1.369	1.516
Equatorial B–H	0.166	0.596	0.700	0.942	1.211	1.327	1.347	1.362	1.405	1.537
B–H bonded to two C's	0.092	0.129	0.416	0.694	0.980	1.196	1.502	2.187	2.217	2.323
Lowest empty MO										
Apical B–H	0.501	0.623	0.714	0.746	0.987	1.099	1.450	1.723	1.799	1.841
Equatorial B–H	0.083	0.200	0.448	0.640	0.951	1.162	1.486	1.576	1.685	1.899
B–H bonded to two C's	0.003	0.205	0.284	0.328	0.423	0.582	0.635	0.837	0.979	1.046

philic or nucleophilic reactions. Table XIV shows the cumulative orbital charge for each type of B starting with the HFMO and moving in the direction of more stable orbitals. Also shown is the subtotal cumulative charge for the empty orbitals of lowest energy (LEMO) moving toward less stable orbitals. No significantly new trends are seen in the relative orders of these partial charges, except in the results from NEMO II, where for the first six HFMO's the cumulative charges (disregarding the sign of the electron) are in the expected order $B_8 > B_4$, B_5, B_7, $B_9 > B_2$, B_3, in agreement with the results from the other methods, but if more than six MO's are included, the order for the NEMO II method reverts to the anomalous order observed in the total Mulliken charges calculated by this method. No new effects are observed from the cumulative framework charges (Table XV).

In summary, we are most inclined to accept the more thoroughly tried methods, the extended Hückel and NEMO I methods. However, all three methods are in agreement when the most polarizable orbitals are considered, and the resulting order of increasing charge (most negative) $B_8 < B_4$ (B_5, B_6, B_7) $< B_2$ (B_3) (most positive) is in agreement with the simple inductive rule[26] that the order from most negative to most positive is B_8 (two C neighbors), B_4 (B_5, B_6, B_7) (one C neighbor), B_2 (B_3) (no C neighbors).

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