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Neutron Diffraction Study of Decaborane[1]

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A three-dimensional neutron diffraction study on an ordered crystal of deuterated decaborane ($^{11}B_{10}{}^2H_{14}$) at $-160°$ has been completed. This is the first investigation of a boron hydride by neutron diffraction, and the accuracy of the hydrogen atom positions is greater than that of any X-ray or electron diffraction study. The molecular structure was found to be consistent with the cap model introduced by Kasper, Lucht, and Harker[3] (space group C2/a-$C_{2h}{}^4$ with a = 14.23 Å, b = 20.26 Å, c = 5.62 Å, and β = 90.10° at $-160°$; Z = 8). The bridging deuterium atoms are in asymmetric positions with respect to the bonding boron atoms in agreement with earlier X-ray investigations. The average values of the B–H bond lengths are 1.180 Å and 1.298, 1.347 Å for the unbridged and bridged hydrogen atoms, respectively. The standard deviations of the bond lengths are 0.004–0.007 Å, and the B–B bond distances and angles are in good agreement with those determined by X-rays. The data indicate that hydrogen–deuterium atom exchange takes place preferentially at the bridged positions. The coherent neutron scattering length for ^{11}B has been refined in an earlier neutron diffraction experiment at room temperature using a crystal exhibiting some disorder. The refined value is $(0.64 \pm 0.01) \times 10^{-12}$ cm.

Introduction

In 1950, Kasper, Lucht, and Harker (KLH)[3] determined the structure of decaborane, $B_{10}H_{14}$, from X-ray diffraction data. The space group was found to be C2/a-$C_{2h}{}^4$ with eight molecules in a unit cell with dimensions a = 14.45 Å, b = 20.88 Å, c = 5.68 Å, and $\alpha = \beta = \gamma = 90°$. The reflections with h and k both odd were diffuse, suggesting a lack of long-range order in the 001 plane. KLH obtained a satisfactory structure on the basis of a disordered model with a smaller cell ($a' = a/2$, $b' = b/2$, $c' = c$). The space group assumed was Pnnm-$D_{2h}{}^{12}$. More recent X-ray investigations of decaborane derivatives[4,5] and reanalysis of the original data[6] on decaborane are in reasonably good agreement regarding B–B bond distances (see Table I); the positions of the hydrogen atoms were not determined to high accuracy in any of these studies.

We have undertaken neutron diffraction studies of both disordered and ordered crystals of decaborane to obtain more accurate hydrogen atom positions and B–H bond lengths. This is the first report of a neutron diffraction study of a boron hydride.

Because of the high absorption cross section of ^{10}B for thermal neutrons, it seemed advisable to prepare decaborane using the isotope ^{11}B. Furthermore, since the deuterium scattering amplitude is greater than that for ^1H, it was felt that substitution of ^1H by ^2H would be helpful in the attainment of high accuracy. Material with the nominal composition $^{11}B_{10}{}^2H_{14}$ was used in the experiments. $^{11}B_{10}{}^2H_{14}$ crystals, prepared by Dr. M. Dettke and Dr. J. Kurzidim, Fritz-Haber-Institut der Max Planck-Gesellschaft, Berlin, were obtained by sublimation (resulting in disordered crystals) or crystallization from ether solution (resulting in ordered crystals). In the case of the disordered crystals a mass spectrometric isotopic analysis of the crystal used for the diffraction measurements gave an isotopic composition of 91.2% ^2H (±0.5%) and 98.4% ^{11}B; in the case of the ordered crystals the isotopic composition was 80.1% ^2H (±0.5%) and 98.4% ^{11}B. The starting material was identical, so there was apparently some H atom exchange in the solution-grown crystals.

A. The Disordered Crystal and Refinement of the Coherent Neutron Scattering Length for ^{11}B

The coherent neutron scattering length for ^{11}B is not well known; values varying from 0.60×10^{-12} to 0.66×10^{-12} cm have been reported.[7,8] Therefore the boron scattering length was included as a parameter in a least-squares refinement using three-dimensional neutron diffraction data taken from a decaborane single crystal with the disordered structure. This crystal was used rather than the ordered one because of the smaller percentage of ^1H.

Data Collection and Processing.—A well-formed colorless crystal 2.5 mm long and 1 mm² in cross section was sealed in a quartz capillary and mounted with the c^* axis parallel to the ϕ axis on a four-circle goniometer at the Brookhaven National Laboratory high-flux beam reactor. Using a neutron wavelength of 1.047 Å and a θ-2θ scan technique, the intensities of 421 independent "sharp" reflections (h and k both even) with a signal-to-background ratio greater than 1:1 were obtained at room temperature. Of these, 165 were measured more than once. For these reflections the agreement factor is $R = 0.06$ ($R = \Sigma |F - \bar{F}|/\Sigma|F|$). No correction was made for absorption. The absorption coefficient based upon the known isotopic composition was $\mu = 3.0$ cm^{-1}. The maximum error introduced in a structure amplitude by neglect of absorption was 7%.

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[3] J. S. Kasper, C. M. Lucht, and D. Harker, *Acta Cryst.*, **3**, 436 (1950).

[4] A. Perloff, *ibid.*, **17**, 332 (1964).

[5] A. Sequeira and W. C. Hamilton, *Inorg. Chem.*, **6**, 128 (1967).

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

[7] T. M. Sabine and B. M. Craven, *Acta Cryst.*, **20**, 214 (1966).

[8] R. E. Donaldson, L. Passel, W. Bartolini, and D. Groves, Report UCRL-12270, Lawrence Radiation Laboratory, 1964.

TABLE I

COMPARISON OF BOND DISTANCES IN $B_{10}H_{14}$, $B_{10}H_{13}(C_2H_5)$, $B_{10}H_{13}I$, AND $^{11}B_{10}D_{14}$ (Å)

Distance	$B_{10}H_{14}$[a]	$B_{10}H_{14}$[b]	$B_{10}H_{13}(C_2H_5)$[c]	$B_{10}H_{13}I$[d]	$^{11}B_{10}{}^2H_{14}$
B1–B2	1.74	1.80	1.79	1.79	1.785
B1–B3	1.76	1.77	1.77	1.78	1.788
B1–B5′	1.74	1.77	1.74	1.77	1.754
B2–B3	1.73	1.72	1.72	1.73	1.715
B2–B4	1.76	1.76	1.77	1.80	1.787
B2–B5	1.81	1.80	1.77	1.77	1.782
B2–B5′	1.79	1.78	1.78	1.78	1.774
B3–B4	1.78	1.77	1.79	1.81	1.762
B4–B5	1.74	1.78	1.76	1.74	1.758
B5–B5′	1.78	1.71	1.78	1.75	1.772
B1–B4′	2.01	2.01	1.96	2.01	1.973
B1–D1(H1)	1.26	1.22	0.97		1.173
B2–D2(H2)	1.29	1.27	1.15		1.177
B3–D3(H3)	1.25	1.28	1.12	1.20	1.182
B4–D4(H4)	1.29	1.35	1.13		1.178
B5–D5(H5)	1.28	1.16	1.00		1.192
B1–D6(H6)	1.34	1.39	1.13		1.298
B3–D6(H6)	1.40	1.43	1.34	1.26	1.355
B4–D7(H7)	1.34	1.25	1.18		1.297
B3–D7(H7)	1.40	1.50	1.25		1.339

[a] Reference 3. [b] Reference 6. [c] Reference 4. [d] Reference 5.

Least-Squares Refinements.—The least-squares refinements were carried out on a CDC 6600 computer using the BNL version of the Busing–Martin–Levy program.[9] The function $\Sigma w(|F_o| - |F_c|)^2$ was minimized, where F_o and F_c are the observed and calculated structure factors. The weights w were assigned in the following way:[10] $w^{1/2} = 1/|F_o|$ if $|F_o| > 4|F_{\min}|$; $w^{1/2} = |F|/(16|F_{\min}|^2)$ if $|F_o| \leqslant 4|F_{\min}|$, where $F_{\min}$ is the smallest observed intensity. The usefulness of this weighting scheme was checked using the program NANOVA (available on request from W. C. Hamilton). The program divided the data into intensity classes and calculated any correlation between the values of $\Delta F/\sigma$ and intensity. In this case $\Delta F/\sigma$ was independent of F, as expected for a proper weighting function. The weighted and unweighted agreement factors

$$R_w = \left[\frac{\Sigma w||F_o| - |F_c||^2}{\Sigma w|F_o|^2}\right]^{1/2}$$

$$R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$$

were used to assess the progress of the refinement.

The least-squares refinement took the structure of KLH as a point of departure. Three cycles of refinement with isotropic temperature factors reduced the values of R_w and R from 0.461 and 0.326 to 0.219 and 0.166.

Anisotropic thermal parameters were now added in the form

$$T_i = \exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$$

Refinement of all parameters except scattering lengths led to $R_w = 0.085$ and $R = 0.068$. Next, the coherent scattering length for B was included as a parameter. In a full-matrix least-squares refinement (with fixed deuterium scattering factors) the refined value for the boron scattering amplitude is $(0.63 \pm 0.01) \times 10^{-12}$ cm. (Any difference in the distribution of ^1H and ^2H on the different sites had no significant effect on the scattering amplitudes for the different hydrogen positions. A refinement of all deuterium scattering amplitudes showed these amplitudes to be equal within the limits of $\pm 0.01 \times 10^{-12}$ cm.) This is an average value, calculated from the refined scattering amplitudes for each boron atom. (The maximum difference between two of these scattering amplitudes was 0.01×10^{-12} cm.) The value is relative only, being based on a deuterium scattering amplitude of 0.66×10^{-12} cm,[11] a hydrogen scattering amplitude of -0.378×10^{-12} cm, and the isotopic composition. Taking into account the small coherent scattering amplitude of ^{10}B (about 0.1×10^{-12} cm) and the isotopic composition of boron, the refined boron scattering amplitude of $(0.63 \pm 0.01) \times 10^{-12}$ cm corresponds to a value of $(0.64 \pm 0.01) \times 10^{-12}$ cm for ^{11}B. [The value of 0.1×10^{-12} cm for the ^{10}B scattering amplitude is derived from the total B coherent scattering length of 0.60 (S. W. Peterson and H. G. Smith, *J. Phys. Soc. Japan*, **17**, 335 (1962)), the value for ^{11}B determined here, and the known isotopic composition.] The *structural* parameters from this refinement did not differ significantly from those for the ordered crystal. Since they were somewhat less precisely determined, they are not quoted here. The structure factors for the common reflections agreed well.

B. The Ordered Crystal

Using the principles of derivative structures (Buer-

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(9) W. R. Busing, K. L. Martin, and H. A. Levy, Report ORNL-TM 305, Oak Ridge National Laboratory, 1962.

(10) L. R. Lavine and W. N. Lipscomb, *J. Chem. Phys.*, **22**, 614 (1954).

(11) T. M. Sabine, P. Coppens, and B. M. Craven, to be submitted for publication.

ger[12]) KLH determined the space group for the ordered structure to be C2/a-C_{2h}^4 (twofold axis in the *c* direction). There are two possible molecular structures for the ordered state derivable from the disordered structure and consistent with this space group: the cap and the crown model (see KLH). According to the structures of $B_{10}H_{13}I$[5] and $B_{10}H_{13}(C_2H_5)$[4] and consistent with the structures of other boron hydrides, the cap model should be correct for decaborane. However, no direct test using X-ray or neutron diffraction methods has previously been made. Electron diffraction data have been shown to be consistent with the cap model.[13]

Data Collection and Processing.—Because rotating-crystal and precession photographs showed no diffuseness of reflections for *h* and *k* both odd (the indication for the disordered structure), the crystal used in this experiment was considered to be well ordered. The crystal (volume 5.7 mm^3) was cut from a bigger one and sealed in a quartz capillary in the presence of dry argon gas. The chunky crystal may be described by the Miller indices (*hkl*) of its bounding planes (not necessarily faces of natural growth) and the perpendicular distances (*d*) of these planes from an arbitrary origin on the surface of the crystal: *h, k, l, d* (cm): −3, 13, 0, 0.13; 2, 1, 0, 0.00; 1, −2, 0, 0.00; −4, −3, 0, 0.118; 0, 0, 1, 0.15; 0, 0, −1, 0.15.

Using the same technique as described in section A (c^* parallel to ϕ, λ 1.073 Å), the intensities of 1333 independent reflections with a signal-to-background ratio greater than 1:1 were obtained at −160 ± 1°. For 729 reflections, measured more than once, the agreement factor on F^2 was $R = 0.06 (R = \Sigma|F^2 - \overline{F^2}|/\Sigma|F|^2)$. The low temperature was achieved by blowing a cold nitrogen gas stream at the crystal using the apparatus ALTA described by Rudman and Godel.[14] The temperature was automatically controlled using a thermocouple as sensor. Using the 2θ values of 87 strong reflections with $2\theta > 60°$ the cell parameters at −160° were determined to be $a = 14.23 \pm 0.02$ Å, $b = 20.26 \pm 0.02$ Å, $c = 5.62 \pm 0.01$ Å, $\alpha = \gamma = 90°$ (fixed), and $\beta = 90.10 \pm 0.1$. The least-squares calculation was carried out using the program CELDIM (available from W. C. H.). The slight difference from 90° of the monoclinic angle is real, as Dierks and Dietrich[15] have found a value of 90.07° at room temperature for a single crystal of $^{11}B_{10}{}^2H_{14}$ from the same preparation using X-ray data.

After applying the usual Lorentz factor, the observed intensities were corrected for absorption. The absorption coefficient based upon the known isotopic composition was $\mu = 3.4$ cm^{-1}. For the extinction correction (applied during the least-square refinement) the approximation introduced by Zachariasen[16] was used. The parameter *g* defined by Zachariasen was refined as described below.

Least-Squares Refinement.—In the least-squares refinement the function $\Sigma w(F_o^2 - F_c^2)^2$ was minimized. F_o and F_c are the observed and calculated structure factors, respectively. To get a proper weighting scheme, the F_o^2 values were divided into 28 intensity classes and the weight for each class was calculated using the program NANOVA as described in section A. Starting with the positional parameters from the disordered structure, these parameters were refined along with isotropic thermal parameters, a scale factor, and the extinction parameter *g* for both the cap and the crown models. Two cycles of refinement led to weighted and unweighted *R* values, R_w and *R*, of 0.099 and 0.116 for the cap model and 0.261 and 0.436 for the crown model, where $R_w = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w F_o^4]^{1/2}$ and $R = \Sigma|F_o^2 - F_c^2|/\Sigma F_o^2$. According to these results the cap model as expected is the right structure model for decaborane. Therefore only this model was used in the final refinement. Taking into account the lack of knowledge about the hydrogen–deuterium distribution, the atomic scattering amplitudes for all seven hydrogen positions were allowed to vary in two cycles of refinement along with the positional parameters, anisotropic thermal parameters, and the extinction parameter *g*. The final *R* values (based on F^2) are $R_w = 0.063$ and $R = 0.095$. The refined scattering factors for the hydrogen–deuterium atoms are listed in Table II. The observed and calculated structure factors (F^2) are presented in Table III and the final positional and thermal parameters are in Table IV. The refined value for the extinction parameter is $g = (1.23 \pm 0.16) \times 10^{-4}$. The extinction correction was in general very small; only a few strong reflections gave rise to a correction factor between 0.83 and 0.98. The refined value for the extinction parameter *g* may be interpreted for the two limiting cases introduced by Zachariasen.[16] In the first case (type I crystals) the extinction is dominated by the mosaic spread. The refined value $g = (1.23 \pm 0.16) \times 10^{-4}$ corresponds to a mosaic spread parameter of 47.4 sec. In the second case (type II crystals), the extinction is dominated by the domain size. Here the calculation led to an average domain size of 1.32×10^{-5} cm.

TABLE II

REFINED SCATTERING AMPLITUDE FOR THE HYDROGEN ATOMS

Atom	Mean scattering factor × 10^{12}, cm	Atom	Mean scattering factor × 10^{12}, cm
D1	0.421 ± 0.01	D5	0.575 ± 0.01
D2	0.502 ± 0.01	D6	0.336 ± 0.01
D3	0.394 ± 0.01	D7	0.354 ± 0.01
D4	0.401 ± 0.01		

Results

The intramolecular distances and bond lengths are listed in Tables V and VI, respectively. The designation of the atoms follows that of KLH and is shown in Figure 1. The B–B bond distances are in good agreement with those found in earlier X-ray studies (see Table I). A comparison of the B–H distances between the neutron

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(12) M. J. Buerger, *J. Chem. Phys.*, **15**, 1 (1947).
(13) C. M. Lucht, *J. Am. Chem. Soc.*, **73**, 2373 (1951).
(14) R. Rudman and J. Godel, *J. Sci. Instr.*, in press.
(15) H. Dierks and H. Dietrich, private communication.
(16) W. H. Zachariasen, *Acta Cryst.*, **23**, 558 (1967).

TABLE III

OBSERVED AND CALCULATED STRUCTURE FACTORS (F^2)

H= -8

K	L	FOBS	FCALC
0	4	156	166
0	6	10	6
2	0	43	46
2	1	76	76
2	2	61	67
2	3	35	33
2	4	211	222
2	5	18	19
2	6	125	150
4	0	135	137
4	1	127	139
4	2	19	19
4	3	300	304
4	4	34	19
4	5	4	13
4	6	28	11
6	0	6	5
6	1	7	4
6	2	38	38
6	3	36	38
6	4	86	92
6	5	6	0
6	6	51	47
8	0	22	12
8	1	19	22
8	2	31	29
8	3	290	321
8	4	10	6
10	0	20	23
10	2	15	17
10	3	4	0
10	4	20	16
10	5	3	4
12	0	25	27
12	1	356	397
12	2	8	2
12	4	10	8
12	5	102	118
14	0	219	239
14	1	69	73
14	2	26	22
14	3	3	4
14	4	4	1
14	5	18	9
16	0	105	105
16	1	28	23
16	2	6	6
16	3	7	7
16	4	22	19
18	0	7	8
18	1	1	2
18	2	16	15
18	3	161	136
18	4	58	47
20	0	11	12
20	1	10	5
20	2	17	7
20	3	51	54
22	0	14	19
22	1	25	24
22	2	5	2

H= -7

K	L	FOBS	FCALC
1	1	138	134
1	2	4	2
1	3	219	233
1	4	33	37
1	5	123	130
1	6	25	23
3	1	250	261
3	2	41	45
3	3	65	57
3	4	18	3
3	5	107	114
3	6	32	27
5	1	8	1
5	3	21	29
5	4	294	305
5	5	27	22
5	6	221	232
7	1	156	152
7	2	11	10
7	3	293	321
7	4	14	4
7	5	144	116
7	6	5	0
9	1	97	106
9	2	81	70
9	3	66	51
9	4	50	58
9	5	12	13
9	6	120	92
11	1	4	3
11	2	4	3
11	3	105	109
11	5	2	0
11	6	16	9
13	1	84	94
13	2	2	0
13	3	84	103
13	4	14	9
13	5	50	38
15	2	39	27
15	3	49	53
15	4	3	3
15	5	52	41
17	1	2	0
17	2	15	15
17	3	23	22
17	4	9	15
19	2	27	27
19	3	47	43
19	4	4	3
21	1	41	44
21	2	9	9
21	3	2	1
23	1	2	2
23	2	4	7

H= -6

K	L	FOBS	FCALC
0	1	103	108
0	3	228	219
0	5	103	98
2	1	444	454
2	2	42	47
2	3	298	261
2	4	27	32
2	5	131	165
2	6	115	131
4	0	2	6
4	1	138	144
4	2	73	85
4	3	31	34
4	4	441	444
4	5	125	152
4	6	108	111
6	0	199	214
6	1	16	20
6	2	6	3
6	3	28	31
6	4	15	8
6	5	24	29
6	6	12	18
8	0	187	231
8	1	11	14
8	2	16	19
8	3	20	32
8	4	8	4
8	5	2	0
8	6	63	57
10	0	3	5
10	1	314	356
10	2	510	542
10	3	45	46
10	4	12	10
10	6	156	148
12	0	353	371
12	1	215	230
12	2	5	3
12	3	26	25
12	4	8	3
12	5	189	186
12	6	98	86
14	0	3	1
14	1	6	6
14	2	72	74
14	3	89	105
14	4	26	35
14	5	4	4
16	0	2	0
16	1	7	5
16	2	26	19
16	3	254	284
16	4	191	202
16	5	2	2
18	0	5	2
18	2	195	219
18	4	63	60
20	0	5	3
20	2	227	240
20	3	173	205
20	4	37	38
22	0	196	195
22	1	6	5
22	2	168	174
22	3	26	28
24	0	250	250
24	1	10	12

H= -5

K	L	FOBS	FCALC
1	1	26	25
1	2	6	6
1	4	25	15
1	5	6	5
1	6	26	16
3	1	28	24
3	2	94	104
3	4	13	12
3	5	35	29
3	6	29	19
5	1	147	134
5	2	7	6
5	3	41	35
5	4	28	27
5	5	39	46
5	6	12	9
7	1	5	6
7	2	61	58
7	3	5	3
7	4	22	23
7	5	2	0
7	6	2	2
9	1	5	6
9	3	105	106
9	4	4	6
9	5	6	3
11	1	37	39
11	2	17	15
11	3	25	25
11	4	79	90
11	5	2	8
11	6	7	4
13	1	63	60
13	2	17	26
13	3	2	1
13	4	9	3
13	5	9	3
15	1	54	48
15	2	29	38
15	3	19	22
15	4	3	2
15	5	12	12
17	1	9	13
17	3	84	58
17	4	7	5
17	5	3	0
19	1	8	4
19	2	9	3
19	3	15	12
19	4	22	34
21	1	10	10
21	2	1	8
21	3	2	0
23	1	43	44
23	2	65	55
25	1	2	0

H= -4

K	L	FOBS	FCALC
0	0	173	165
0	2	2	0
0	4	157	155
0	6	349	320
2	0	11	8
2	1	220	236
2	2	191	187
2	3	25	25
2	4	63	54
2	5	9	10
2	6	140	145
2	7	65	86
4	0	397	434
4	1	119	112
4	2	3	3
4	3	30	31
4	4	59	54
4	6	127	129
6	0	503	529
6	1	1	0
6	3	77	72
6	4	336	351
6	5	27	30
6	6	9	7
8	0	68	62
8	1	78	76
8	2	88	78
8	3	207	215
8	4	13	11
8	5	11	15
8	6	43	41
10	0	56	43
10	1	11	13
10	2	45	42
10	3	67	69
10	4	310	309
10	5	2	0
10	6	2	0
12	0	181	217
12	2	118	112
12	3	14	12
12	4	27	25
12	5	9	3
12	6	147	152
14	0	222	251
14	1	96	89
14	2	71	61
14	3	7	4
14	4	113	112
14	5	32	31
16	0	212	224
16	1	419	437
16	2	18	17
16	3	112	121
16	4	95	99
16	5	101	100
18	0	153	156
18	1	2	1
18	2	143	146
18	3	410	407
18	4	104	110
18	5	69	72
20	0	17	8
20	1	122	111
20	2	334	318
20	3	89	89
20	4	111	108
22	0	19	17
22	1	17	15
22	2	168	153
22	3	178	159
24	0	171	152
24	1	16	20
24	2	31	25

H= -3

K	L	FOBS	FCALC
1	1	84	96
1	2	3	3
1	3	3	2
1	4	5	1
1	5	107	103
1	7	265	273
3	1	14	10
3	2	48	46
3	3	36	34
3	4	30	21
3	5	8	11
3	6	2	0
3	7	52	61
5	2	9	7
5	3	36	22
5	4	5	5
5	5	3	5
5	6	20	21
5	7	33	20
7	1	25	28
7	2	28	47
7	3	32	31
7	4	9	19
7	5	5	3
7	6	9	4
9	1	27	34
9	2	7	4
9	3	8	10
9	4	161	155
9	5	45	51
9	6	137	126
11	1	134	145
11	2	63	72
11	3	37	41
11	4	32	21
11	5	8	5
11	6	5	7
13	1	31	35
13	2	19	11
13	3	124	147
13	4	81	115
13	5	45	64
13	6	114	81
15	1	37	28
15	2	112	111
15	3	57	73
15	4	14	11
15	5	4	0
17	1	19	13
17	2	25	13
17	4	3	2
17	5	5	4
19	2	1	0
19	3	5	0
21	1	11	21
21	2	1	0
21	3	122	130
23	2	7	5
23	3	35	40
25	1	64	68

H= -2

K	L	FOBS	FCALC
0	1	174	176
0	3	24	19
0	5	752	688
0	6	2	0
0	7	28	21
2	0	373	401
2	2	7	5
2	3	45	41
2	4	87	84
2	5	56	44
2	6	13	6
2	7	2	0
4	0	17	18
4	1	5	3
4	2	374	379
4	3	124	149
4	4	155	156
4	5	2	2
4	6	4	4
4	7	36	34
6	0	136	147
6	1	8	6
6	2	45	56
6	3	217	221
6	4	5	9
6	5	184	195
8	0	428	450
8	1	2	2
8	2	38	41
8	3	4	4
8	4	258	252
8	5	103	113
8	6	52	42
10	0	118	126
10	1	38	34
10	3	40	39
10	4	106	102
10	5	11	22
10	6	10	13
12	0	120	136
12	1	25	27
12	2	27	0
12	3	53	53
12	4	203	194
12	5	3	4
12	6	95	90
14	0	130	116
14	1	131	139
14	2	177	196
14	3	22	14
14	4	3	0
14	5	24	13
14	6	29	25
16	0	156	162
16	1	4	7
16	2	122	129
16	3	17	12
16	4	49	40
18	0	144	168
18	1	153	165
18	2	58	56
18	4	16	9
18	5	108	111
20	0	10	8
20	1	2	1
20	2	13	10
20	3	2	1
20	4	8	6
22	0	4	4
22	1	81	72
22	2	12	16
22	3	32	40
24	0	117	125
24	1	5	4
24	2	47	53
26	0	28	29

H= -1

K	L	FOBS	FCALC
1	2	140	142
1	4	8	10
1	6	5	1
1	7	18	7
3	1	18	26
3	2	15	8
3	3	23	28
3	4	16	11
3	7	30	25
5	1	1	1
5	2	14	21
5	3	12	5
5	4	1	0
5	5	28	21
5	6	6	8
5	7	87	94
7	1	2	2
7	2	51	36
7	3	116	97
7	5	18	9
7	6	9	9
7	7	47	56
9	1	25	26
9	2	41	47
9	3	24	16
9	4	9	9
9	5	4	2
9	6	11	16
11	1	6	7
11	2	10	9
11	3	78	84
11	4	33	27
11	5	19	23
11	6	54	44
13	1	44	42
13	2	4	7
13	3	37	49
13	4	23	20
13	5	14	4
15	1	29	37
15	2	24	11
15	3	159	181
15	4	27	31
15	5	14	6
17	1	6	4
17	2	15	7
17	3	19	19
17	4	12	13
17	5	3	3
19	1	4	8
19	2	20	15
19	3	7	9
19	4	17	8
19	5	4	6
21	1	1	2
21	2	16	15
21	3	11	6
21	4	29	30
23	1	49	55
23	2	9	2
23	3	51	50
25	1	5	7
26	2	17	18

H= 0

K	L	FOBS	FCALC
0	2	10	3
0	4	840	850
0	6	142	163
2	1	394	408
2	3	9	10
2	5	400	404
2	7	78	75
4	0	298	285
4	2	836	851
4	4	69	68
4	6	4	4
6	1	16	10
6	3	473	447
6	5	323	324
6	7	53	51
8	0	607	618
8	2	2	1
8	4	301	300
8	6	19	16
10	1	42	43
10	3	7	5
10	5	30	46
12	0	371	379
12	2	668	649
12	4	111	91
12	6	21	17
14	1	12	13
14	3	161	144
14	5	134	114
16	0	588	590
16	2	84	83
16	4	9	1
18	1	176	169
18	5	57	40
20	0	107	106
20	2	11	11
20	4	310	310
22	1	110	116
22	3	570	579
24	0	75	69
24	2	12	14
26	1	68	81

H= 1

K	L	FOBS	FCALC
1	1	54	63
1	2	5	1
1	4	3	2
1	5	7	6
1	6	2	0
1	7	16	8
3	1	5	4
3	2	24	26
3	3	2	2
3	4	2	2
3	5	15	14
3	6	5	3
3	7	53	52
5	1	3	2
5	2	24	26
5	3	108	113
5	4	21	9
5	7	57	85
7	1	4	4
7	2	85	85
7	3	10	8
7	5	9	13
7	6	4	15
7	7	111	100
9	1	4	5
9	2	42	30
9	3	6	3
9	4	2	7
9	5	17	19
9	6	19	28
11	1	48	47
11	2	6	1
11	3	34	32
11	4	21	25
11	5	2	0
11	6	6	5
13	1	16	18
13	2	2	0
13	3	236	239
13	4	26	37
13	5	14	16
13	6	48	44
15	1	22	30
15	2	1	2
15	3	39	45
15	4	4	1
15	5	7	5
17	1	39	37
17	2	23	20
17	3	39	30
17	4	17	18
17	5	4	0
19	1	3	0
19	2	37	38
19	3	9	1
19	4	45	38
19	5	2	1
21	1	11	7
21	2	2	6
21	3	38	46
21	4	13	2
23	1	5	5
23	3	11	20
25	1	66	71

H= 2

K	L	FOBS	FCALC
0	1	174	176
0	3	24	19
0	5	628	688
0	7	24	21
2	0	430	385
2	1	2	2
2	2	11	5
2	3	52	41
2	4	110	94
2	5	47	44
2	6	14	6
2	7	4	0
4	0	25	18
4	1	6	3
4	2	407	375
4	3	144	148
4	4	174	155
4	5	5	2
4	6	7	4
4	7	20	34
6	0	135	146
6	1	8	5
6	2	54	56
6	3	235	221
6	4	10	9
6	5	187	194
6	6	3	0
6	7	4	7
8	0	457	447
8	2	41	41
8	3	6	4
8	4	237	252
8	5	113	114
8	6	55	42
10	0	134	126
10	1	37	34
10	2	1	1
10	3	33	39
10	4	133	152
10	5	20	24
10	6	22	13
12	0	127	136
12	1	23	27
12	2	9	0
12	3	48	53
12	4	202	198
12	5	7	4
12	6	94	90
14	0	96	116
14	1	131	139
14	2	220	196
14	3	16	14
14	5	13	13
14	6	38	25
16	0	138	162
16	1	8	7
16	2	132	129
16	3	13	14
16	4	41	40
16	5	1	1
18	0	188	168
18	1	157	165
18	2	49	56
18	4	9	9
18	5	111	111
20	0	13	6
20	1	1	1
20	2	9	10
20	3	4	1
20	4	8	6
22	0	8	4
22	1	73	72
22	2	19	16
22	3	41	40
24	0	126	125
24	1	8	4
24	2	54	53
26	0	30	29

H= 3

K	L	FOBS	FCALC
1	1	4	3
1	2	15	13
1	3	6	6
1	4	69	69
1	5	10	2
1	6	3	0
3	1	34	36
3	2	11	12
3	3	7	3
3	4	3	1
3	5	55	51
3	6	11	2
3	7	169	137
5	2	82	83
5	3	48	43
5	4	2	0
5	5	8	11
5	6	4	2
5	7	114	97
7	1	14	15
7	2	4	0
7	3	14	10
7	4	67	59
7	5	10	10
7	6	70	71
9	1	116	108
9	2	2	0
9	3	40	26
9	4	58	29
9	5	1	1
9	6	16	5
11	1	34	38
11	2	3	0
11	3	115	98
11	4	154	184
11	5	54	77
11	6	139	131
13	1	111	93
13	2	162	156
13	3	74	70
13	4	32	13
13	5	16	5
13	6	12	9
15	1	42	29
15	2	44	36
15	3	52	61
15	4	23	33
15	5	45	30
17	1	4	2
17	2	35	23
17	3	30	31
17	4	3	9
17	5	1	1
19	1	3	0
19	2	12	8
19	3	60	69
19	4	2	1
21	2	16	8
21	3	23	19
21	4	3	3
23	1	61	64
23	3	84	95
25	1	1	3

H= 4

K	L	FOBS	FCALC
0	0	174	165
0	2	2	0
0	4	118	155
0	6	298	320
2	0	12	8
2	1	246	233
2	2	193	186
2	3	23	25
2	4	64	56
2	5	4	10
2	6	149	144
2	7	84	85
4	0	462	424
4	1	121	111
4	3	39	31
4	4	69	54
4	6	147	129
6	0	570	519
6	3	85	72
6	4	378	349
6	5	30	30
6	6	8	7
8	0	67	62
8	1	85	76
8	2	91	78
8	3	220	214
8	4	6	11
8	5	17	15
8	6	64	41
10	0	45	43
10	1	9	13
10	2	43	42
10	3	68	69
10	4	313	308
10	5	3	0
12	0	225	217
12	2	112	112
12	3	11	12
12	4	40	25
12	5	5	3
12	6	183	152
14	0	277	251
14	1	98	89
14	2	66	61
14	3	8	4
14	4	113	112
14	5	36	31
16	0	224	222
16	1	461	435
16	2	16	17
16	3	114	121
16	4	99	99
16	5	102	100
18	0	161	156
18	1	2	1
18	2	148	146
18	3	409	406
18	4	105	110
18	5	89	72
20	0	9	8
20	1	117	111
20	2	311	317
20	3	101	89
20	4	99	106

H= 5

K	L	FOBS	FCALC
1	1	25	57
1	2	7	18
1	5	14	46
3	1	90	137
3	2	1	0
3	4	12	22
3	5	20	35
3	6	30	14
5	1	3	1
5	2	85	117
5	3	3	5
5	5	6	10
5	6	9	15
7	1	36	28
7	2	8	6
7	3	83	96
7	4	22	21
7	5	16	24
7	6	3	4
9	1	21	24
9	2	14	18
9	3	8	7
9	4	100	88
11	1	58	54
11	2	14	12
11	3	29	35
11	4	4	0
11	6	16	0
13	1	56	48
13	2	27	31
13	3	37	43
13	4	18	26
13	5	14	12
15	1	34	31
15	2	22	15
15	3	49	45
15	4	23	5
17	1	38	29
17	2	6	12
17	3	3	0
17	5	13	10
19	1	7	10
19	2	2	3
19	3	18	20
19	4	11	7
21	1	5	4
21	2	37	41
21	3	27	30

H= 6

K	L	FOBS	FCALC
0	1	101	108
0	3	205	219
0	5	94	98
2	1	501	450
2	2	47	47
2	3	730	740
2	4	33	32
2	5	154	165
2	6	138	131
4	0	7	6
4	1	160	144
4	2	73	86
4	3	40	74
4	4	438	441
4	5	169	152
4	6	95	111
6	0	218	213
6	1	21	20
6	2	2	3
6	3	22	31
6	4	11	8
6	5	30	20
6	6	23	18
8	0	242	229
8	1	14	16
8	2	18	19
8	3	41	32
8	4	13	4
8	5	4	0
8	6	68	57
10	0	4	5
10	1	382	354
10	2	572	537
10	3	43	46
10	4	10	10
10	6	140	148
12	0	378	369
12	1	235	229
12	2	2	3
12	3	29	25
12	4	14	3
12	5	202	185
12	6	96	86
14	0	4	1
14	2	81	74
14	3	115	105
14	4	34	35
14	5	4	4
16	1	5	5
16	2	25	19
16	3	311	283
16	4	197	201
18	0	4	2
18	2	211	219
18	4	60	60
20	0	4	3
20	1	2	1
20	2	268	239
20	3	215	204
20	4	40	38
22	1	4	5
22	2	189	174
22	3	23	38

H= 7

K	L	FOBS	FCALC
1	1	54	44
1	2	57	57
1	3	11	5
1	4	3	2
1	5	17	17
1	6	28	48
3	1	64	57
3	2	8	4
3	3	149	136
3	4	258	229
3	5	67	79
3	6	147	153
5	1	326	314
5	2	29	27
5	3	231	185
5	4	13	4
5	5	151	170
5	6	13	7
7	1	35	37
7	2	35	35
7	3	13	2
7	4	194	186
7	5	6	0
7	6	201	196
9	1	24	17
9	2	3	1
9	3	291	296
9	4	4	2
9	5	60	79
9	6	2	4
11	1	133	134
11	2	42	36
11	3	91	102
11	4	8	13
11	5	31	33
13	1	11	10
13	2	17	14
13	3	2	0
13	4	7	1
13	5	22	22
15	1	31	31
15	2	15	15
15	3	51	47
15	4	18	16
15	5	34	29
17	2	40	37
17	3	106	89
17	4	10	4
19	1	15	14
19	3	4	2
21	1	3	1
21	2	15	16
21	3	6	4
23	1	56	62
23	2	27	14

H= 8

K	L	FOBS	FCALC
0	4	157	166
0	6	11	6
2	0	52	46
2	1	70	76
2	2	66	67
2	3	31	33
2	4	216	221
2	5	21	19
2	6	185	150
4	0	144	137
4	1	135	139
4	2	18	19
4	3	303	307
4	4	14	19
4	5	7	13
4	6	12	11
6	0	7	5
6	1	3	4
6	2	40	38
6	3	31	38
6	4	95	92
6	5	1	0
6	6	60	47
8	0	25	12
8	1	28	22
8	2	39	29
8	3	382	319
8	4	4	6
8	6	3	4
10	0	29	23
10	1	1	0
10	2	12	16
10	3	1	0
10	4	22	16
10	5	6	4
12	0	35	27
12	1	442	395
12	2	5	2
12	4	14	8
12	5	127	117
14	0	252	239
14	1	70	73
14	2	18	22
14	4	4	1
16	0	117	105
16	1	31	23
16	2	4	6
16	3	10	7
16	4	26	19
18	0	11	8
18	1	2	2
18	2	13	15
18	3	124	136
20	0	9	12
20	1	7	5
20	2	8	7

H= 9

K	L	FOBS	FCALC
1	1	5	3
1	3	162	160
1	4	31	18
1	5	59	45
1	6	7	4
3	1	151	135
3	2	4	5
3	3	3	1
3	4	80	80
3	5	19	12
3	6	64	86
5	1	7	1
5	2	35	44
5	3	1	0
5	4	146	147
5	5	1	1
5	6	21	28
7	1	148	140
7	2	21	13
7	3	47	32
7	4	4	2
7	5	15	4
9	1	10	9
9	2	233	221
9	3	41	38
9	4	19	31
9	5	33	30
11	1	45	47
11	2	5	0
11	3	29	26
11	4	27	28
11	5	12	12
13	1	2	2
13	2	34	50
13	3	6	3
13	4	19	12
13	5	29	23
15	1	31	43
15	2	10	4
15	3	28	21
15	4	24	25
17	1	42	38
17	2	3	2
17	4	10	13
19	1	2	0
19	3	31	14

H= 10

K	L	FOBS	FCALC
0	1	51	43
0	2	2	0
0	3	425	418
0	5	11	14
2	0	173	168
2	1	271	265
2	2	17	16
2	3	35	30
2	4	21	8
2	5	17	22
2	6	40	40
4	0	2	1
4	1	305	318
4	2	252	258
4	3	6	4
4	4	11	5
4	5	256	254
6	0	281	292
6	1	47	54
6	2	5	2
6	3	40	52
6	4	8	2
6	5	7	3
8	0	36	39
8	1	77	69
8	2	20	17
8	3	78	78
8	4	429	413
8	5	2	0
10	0	102	96
10	1	30	20
10	2	109	94
10	3	158	160
10	4	100	87
10	5	25	24
12	0	38	51
12	1	201	195
12	2	3	2
12	3	8	6
12	4	42	40
12	5	253	256
14	0	460	453
14	1	102	98
14	4	43	53
16	0	235	235
16	2	15	22
16	3	338	324

H= 11

K	L	FOBS	FCALC
1	1	29	28
1	2	131	144
1	4	5	9
1	5	4	2
3	1	13	13
3	3	9	12
3	4	2	0
3	5	47	54
5	1	313	300
5	3	19	19
5	4	12	12
5	5	38	19
7	1	14	11
7	2	6	5
7	3	63	44
7	4	27	23
7	5	15	14
9	1	119	128
9	2	2	1
9	3	41	36
11	2	11	14
11	3	12	15
11	4	22	39
13	1	2	0
13	2	5	2
13	3	16	15
13	4	2	2
15	2	5	1
15	3	47	65

H= 12

K	L	FOBS	FCALC
0	0	55	37
0	2	124	138
0	4	61	56
2	0	3	1
2	2	14	11
2	3	52	46
2	4	8	7
2	5	2	7
4	0	116	104
4	1	34	40
4	3	63	56
4	4	101	113
4	5	4	1
6	0	51	53
6	1	35	29
6	2	10	8
6	3	6	0
6	4	244	242
6	5	77	94
8	0	6	3
8	1	36	49
8	2	12	13
8	3	403	398
8	4	7	2
8	5	23	35
10	0	6	3
10	1	3	3
10	2	20	24
10	3	33	29
10	4	171	176
12	0	146	124
12	1	125	110
12	3	10	5
12	4	65	54
14	0	457	453
14	1	3	4
14	2	36	35
14	3	98	98
16	0	21	33
16	1	137	137
16	2	67	42
16	3	16	17
18	0	66	72
18	1	118	99
18	2	45	37
20	0	98	116

H= 13

K	L	FOBS	FCALC
1	1	96	107
1	2	6	3
1	3	24	15
1	4	1	0
1	5	16	12
3	1	3	0
3	2	10	10
3	3	3	6
3	4	18	18
3	5	51	42
5	1	14	14
5	2	31	37
5	3	62	47
5	4	19	10
5	5	5	9
7	1	18	18
7	2	15	13
7	3	25	28
7	4	2	0
9	1	49	48
9	2	38	41
9	3	35	46
9	4	32	30
11	1	71	84
11	2	2	2
11	3	3	3
11	4	2	1
13	1	88	68
13	2	49	49
13	3	30	18
15	1	120	124
15	2	3	3
15	3	34	32
17	1	2	0
17	2	25	28

H= 14

K	L	FOBS	FCALC
0	1	258	258
0	3	6	3
2	0	550	550
2	1	142	136
2	2	3	0
2	3	9	8
2	4	23	17
4	0	305	308
4	1	9	7
4	2	6	2
4	3	112	91
4	4	162	142
6	0	56	53
6	1	64	65
6	2	70	59
6	3	54	48
6	4	51	55
8	0	119	125
8	1	12	13
8	2	12	10
8	3	48	33
8	4	44	45
10	0	114	125
10	1	14	23
10	2	2	0
10	3	12	18
12	0	20	15
12	1	17	21
12	3	42	28
14	0	10	4
14	1	16	9
14	2	94	84
16	0	56	53
16	1	15	10

H= 15

K	L	FOBS	FCALC
1	1	20	9
1	2	5	3
1	4	25	13
3	1	105	114
3	2	10	9
5	1	92	100
5	2	56	51
5	3	4	2
5	4	6	3
7	1	6	9
7	2	32	33
7	3	15	11
9	1	30	46
9	2	14	19
9	3	17	15
11	1	11	9
11	2	34	32
11	3	22	13
13	1	11	9
13	2	21	13
15	1	49	63

H= 16

K	L	FOBS	FCALC
0	0	784	786
0	2	91	95
2	0	131	130
2	1	30	41
2	2	32	26
2	3	80	104
4	0	3	0
4	1	10	6
4	2	116	112
4	3	76	75
6	0	168	160
6	1	82	111
6	2	305	312
6	3	162	156
8	0	131	135
8	1	10	11
8	2	100	109
10	0	143	161
10	2	17	18
12	0	10	8
12	1	1	1

H= 17

K	L	FOBS	FCALC
1	1	141	152
3	1	62	63
3	2	35	34
5	1	12	12
5	2	16	9
7	1	256	258
7	2	27	29
9	1	23	24

TABLE IV

ATOMIC PARAMETERS[a]

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
B1	0.0336 (2)	0.3317 (1)	0	15 (1)	8 (1)	90 (6)	−2 (1)	0	0
B2	0.0993 (2)	0.2738 (2)	0.1738 (5)	13 (1)	8 (1)	77 (8)	1 (1)	−5 (3)	−6 (2)
B3	0.1192 (2)	0.2778 (2)	−0.1277 (5)	11 (2)	12 (1)	95 (8)	−3 (1)	7 (3)	−1 (2)
B4	0.0976 (2)	0.1998 (1)	0	12 (1)	7 (1)	104 (7)	0 (1)	0	0
B5	0.0198 (2)	0.2086 (1)	0.2407 (5)	16 (1)	7 (1)	77 (7)	1 (1)	3 (2)	0 (1)
D1	0.0456 (3)	0.3889 (2)	0	28 (2)	9 (1)	251 (16)	0 (1)	0	0
D2	0.1591 (3)	0.2874 (2)	0.3093 (7)	18 (2)	17 (1)	178 (14)	4 (1)	−8 (4)	−10 (3)
D3	0.1853 (4)	0.2946 (3)	−0.2395 (6)	19 (3)	18 (2)	207 (19)	−6 (2)	17 (7)	10 (5)
D4	0.1565 (3)	0.1590 (2)	0	15 (2)	12 (1)	237 (15)	4 (1)	0	0
D5	0.0353 (3)	0.1747 (2)	0.4097 (6)	37 (2)	13 (1)	165 (11)	1 (1)	0 (4)	15 (3)
D6	0.0452 (4)	0.3117 (3)	−0.2176 (5)	21 (4)	12 (2)	64 (18)	−3 (2)	−13 (6)	0 (3)
D7	0.0883 (4)	0.2205 (2)	−0.2172 (5)	20 (3)	13 (2)	75 (16)	−4 (2)	−2 (5)	−8 (3)

[a] The coordinates are expressed as fractions of the cell parameters. The β_{ij}'s are multiplied by 10^4. Estimated standard deviations in the last significant figure are in parentheses.

TABLE V

INTRAMOLECULAR DISTANCES AND THEIR ESD'S FOR $^{11}B_{10}D_{14}$ (80.1% D)

Atoms	Distances, Å	σ, Å
	Interatomic Distances	
B1–B2	1.785	0.005
B1–B3	1.788	0.005
B1–B5′	1.754	0.004
B2–B3	1.715	0.004
B2–B4	1.787	0.005
B2–B5	1.782	0.005
B2–B5′	1.774	0.005
B3–B4	1.762	0.005
B4–B5	1.758	0.004
B5–B5′	1.772	0.006
B4′–B1	1.973	0.004
	Unbridged Hydrogen Bonds	
B1–D1	1.173	0.005
B2–D2	1.177	0.006
B3–D3	1.182	0.007
B4–D4	1.178	0.005
B5–D5	1.192	0.004
	Bridged Hydrogen Bonds	
B1–D6	1.298	0.005
B3–D6	1.355	0.007
B4–D7	1.297	0.005
B3–D7	1.339	0.007

and X-ray studies is not so useful because of the low accuracy of the X-ray results. Nevertheless, this study confirms the slight *but highly significant* asymmetry in the positions of the bridging hydrogen atoms H6 and H7. These atoms are further away from the terminal boron B3 than from B1 and B4. The average values of 1.180 Å for the unbridged B–H distance and 1.298, 1.347 Å for the bridged B–H distances are in good agreement with those found in pentaborane[17] (1.20 and 1.35 Å for the unbridged and bridged B–H distances, respectively). Table VII gives the shortest nonbonded intramolecular and the shortest intermolecular distances, respectively. These values also agree with those found in pentaborane. The bond angles in the boron framework agree with those found in $B_{10}H_{13}I$.[5] The folding angle

TABLE VI

INTRAMOLECULAR BOND ANGLES

Atoms	Angle, deg	σ, deg
B2–B1–B5′	60.2	0.2
B2–B1–D1	125.0	0.3
B3–B1–B2	57.4	0.2
B3–B1–B4′	116.6	0.2
B3–B1–D1	120.3	0.3
B3–B1–D6	49.0	0.3
B3–B1–B5′	108.8	0.2
B4′–B1–D1	117.2	0.3
B4′–B1–B5′	55.8	0.2
B5′–B1–D1	121.5	0.2
D1–B1–D6	106.8	0.3
B1–B2–B3	61.4	0.2
B1–B2–B5′	59.0	0.2
B1–B2–D2	125.3	0.4
B3–B2–B4	60.4	0.3
B3–B2–D2	120.7	0.4
B4–B2–B5	59.0	0.2
B4–B2–D2	124.0	0.4
B5–B2–B5′	59.8	0.2
B5–B2–D2	119.5	0.4
B5′–B2–D2	120.2	0.4
B1–B3–B2	61.2	0.3
B1–B3–D3	125.5	0.5
B1–B3–B4	105.3	0.2
B1–B3–D6	46.3	0.3
B2–B3–B4	61.8	0.3
B2–B3–D3	132.0	0.4
B4–B3–D3	127.9	0.5
B4–B3–D7	47.0	0.3
D3–B3–D6	106.0	0.5
D3–B3–D7	108.1	0.5
B1′–B4–B5	55.6	0.2
B1′–B4–D4	116.5	0.3
B1′–B4–D7	90.6	0.3
B2–B4–B3	57.8	0.2
B2–B4–B5	60.3	0.2
B2–B4–D4	125.5	0.3
B2–B4–D7	104.0	0.3
B3–B4–D4	120.3	0.3
B3–B4–D7	49.1	0.3
B5–B4–D4	121.3	0.2
D4–B4–D7	107.3	0.3
B1′–B5–B4	68.4	0.2
B1′–B5–D5	115.3	0.3
B2–B5–B4	60.6	0.2
B2–B5–B5′	59.9	0.2
B2–B5–D5	118.8	0.3
B4–B5–D5	116.1	0.3
B5′–B5–D5	127.2	0.2

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(17) W. F. Dulmage and W. N. Lipscomb, *Acta Cryst.*, **5**, 260 (1952).

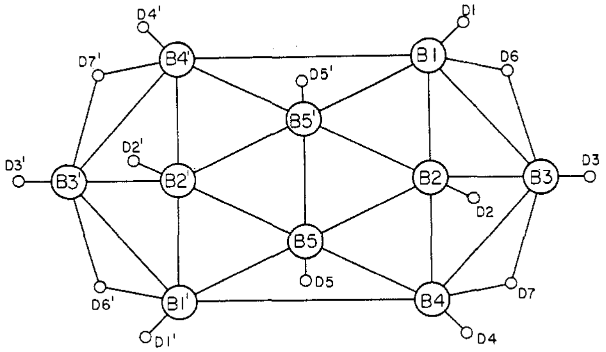

Figure 1.—Topological diagram of decaborane.

TABLE VII

SHORTEST NONBONDED INTRAMOLECULAR AND INTERMOLECULAR DISTANCES

Atoms	Nonbonded intramolecular distances, Å
D6–D1	1.985 (0.006)
D6–D3	2.029 (0.008)
D6–D7	1.949 (0.008)
D6–D7′	2.009 (0.009)
D7–D3	2.043 (0.008)
D7–D4	1.994 (0.006)
Atoms	**Shortest intermolecular distances, Å**
D7–D5[a]	2.461 (0.006)
B3–D2[a]	3.300 (0.006)

[a] In each case the second atom is in the molecule related to the basic molecule by a translation of −**c**.

of the decaborane molecule between the two planes B5–B5′–B1 and B5–B5′–B1′ is 71.8 ± 0.3°, compared to the 76° found by KLH. This difference may be partly due to the fact that the data were collected at different temperatures. A calculation of this angle using the data from the crystal with disordered structure (at room temperature) gave 73.2 ± 0.6°.

According to Table II, hydrogen atoms are preferentially at bridge positions. Using the neutron coherent scattering lengths for ^{2}H and ^{1}H, 0.66 × 10^{-12} and −0.378 × 10^{-12} cm, respectively, the average percentage is 30 and 19% ^{1}H at bridged and unbridged positions, respectively. (Nevertheless, in the tables and figures we refer to the hydrogen atoms as D.) The crystal was grown in ethereal solution from material with an average isotopic composition of 8.8% ^{1}H and 91.2% ^{2}H. Apparently during the crystallization a deuterium–hydrogen exchange took place. According to the isotopic composition calculated from the refined scattering lengths of the hydrogen–deuterium atoms, such exchange takes place preferentially at the bridge position. This is in agreement with nmr experiments[18] and charge distribution calculations.[19] The significant differences in the refined scattering factors for the unbridged hydrogen positions (see Table II) are also qualitatively understandable by considering the charge distributions for the boron atoms in decaborane as calculated by Lipscomb[19] and listed in Table VIII. From these values one would expect that the strongest B–H bonds are B5–D5 and B2–D2. Therefore the ^{2}H–^{1}H exchange should be least at these positions. The opposite is true for the B3–D3 bond. The strengths of the B1–D1 and B4–D4 bonds are equal and between those for B3–D3 and B2–D2. The refined scattering factors listed in Table II confirm these expectations.

TABLE VIII

CHARGE DISTRIBUTIONS IN DECABORANE[19]

Atom	Charge
B1	0.12
B2	−0.10
B3	0.33
B3	0.12
B5	−0.04

In Table IX the rms components of the thermal motion (in Å) along the principal axes of the thermal displacement ellipsoids are listed. These values have been calculated from the thermal parameters listed in Table II, using the BNL version of the ORFFE program.[20] For the unbridged deuterium atoms, the direction of maximum vibration is perpendicular to the B–H bonds as expected; for the bridged deuterium atoms the maximum thermal displacement is in a plane parallel to the B–B bond belonging to the bridge. Normal to this plane the vibrations are essentially isotropic. One can see this clearly in Figure 2 which shows a stereoview of the decaborane molecule. Figure 3 shows a stereoview of the unit cell of decaborane. In order to give a clear view of the packing of the molecules, the hydrogen atoms are not included.

TABLE IX

ATOMIC THERMAL DISPLACEMENTS[a]

Atom	r_1	σ	r_2	σ	r_3	σ
B1	11.5	0.6	12.0	0.4	13.9	0.5
B2	10.1	0.7	10.9	0.8	13.9	0.7
B3	9.6	0.9	12.7	0.6	16.2	0.6
B4	11.2	0.6	12.4	0.4	12.9	0.4
B5	10.9	0.5	11.7	0.6	13.2	0.6
D1	13.8	0.8	17.0	0.8	20.0	0.6
D2	11.9	1.1	16.8	0.8	19.7	0.8
D3	11.0	1.5	18.8	1.1	20.8	1.1
D4	11.3	1.1	16.4	0.8	19.5	0.6
D5	13.4	0.8	18.6	0.6	19.6	0.7
D6	8.9	1.8	14.0	1.3	16.9	1.3
D7	9.9	1.4	13.5	1.3	17.6	1.1

[a] The rms amplitudes r_1, r_2, r_3 are parallel to the principal axes of the thermal displacement ellipsoid (units: Å × 10^{-2}).

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(18) I. Shapiro, M. Lustig, and R. E. Williams, *J. Am. Chem. Soc.*, **81**, 838 (1959); R. E. Williams, *Inorg. Chem.*, **4**, 1504 (1965).

(19) W. N. Lipscomb, "Boron Hydrides," W. A. Benjamin, Inc., New York, N. Y., 1963.

(20) W. R. Busing, K. L. Martin, and H. A. Levy, Report ORNL-TM-306, Oak Ridge National Laboratory, 1964.

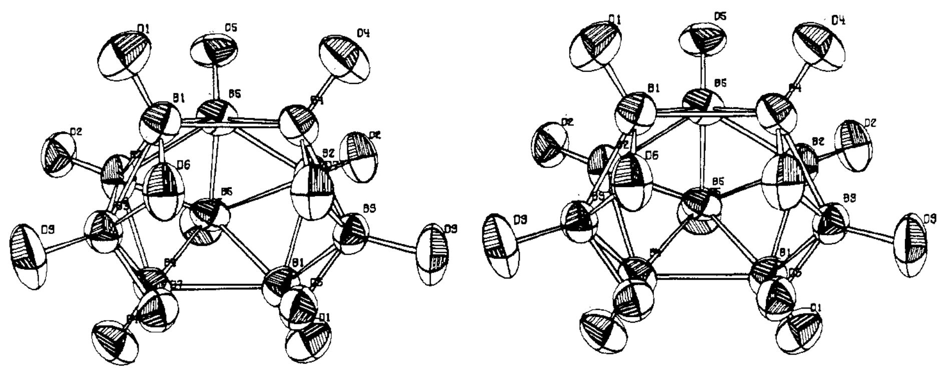

Figure 2.—Stereoview of the decaborane molecule. The ellipsoids indicate the magnitudes of thermal vibration.

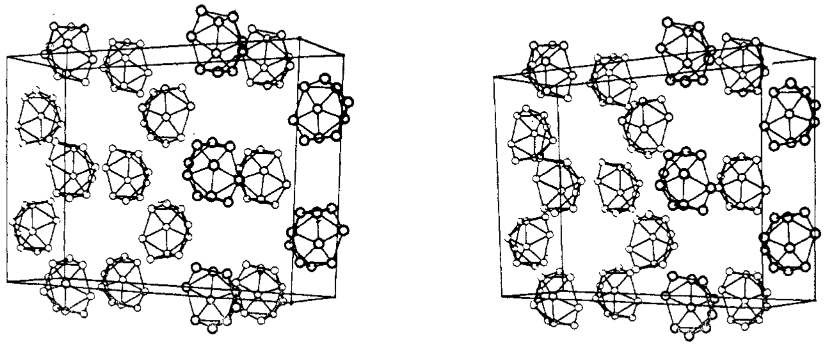

Figure 3.—Packing diagram of the decaborane unit cell (stereoview). The hydrogen atoms are not included.

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Metal Complexes of the Dibenzamido Anion

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Several new metal complexes of the dibenzamido anion, $M(C_6H_5CONCOC_6H_5)_n$, where M = Be(II), Al(III), Fe(III), Cu(II), and Hg(II), have been synthesized. The compounds were characterized by elemental analysis and from their infrared spectra. A comparison of the electronic properties of some isoelectronic and isostructural dibenzamido and dibenzoylmethano complexes is presented.

Introduction

Tris(dibenzamido)chromium(III), $Cr(dba)_3$, and tris(diacetamido)chromium(III), $Cr(daa)_3$, were reported recently as the first well-characterized complexes of diacylamide anions.[1] Further attempts to obtain other new diacetamido–metal complexes have been generally unsuccessful. However, several new complexes of the dibenzamido anion have been isolated and characterized and will be reported on at this time.[2]

Experimental Section

Reagents.—Anhydrous beryllium chloride was obtained from Alfa Inorganics, Inc. Anhydrous chromium(III) chloride[3] and iron(III) chloride[4] were prepared in the laboratory. Anhydrous aluminum chloride (Baker and Adamson) was sublimed before use. Copper(II) acetate monohydrate and mercury(II) acetate were purchased from the J. T. Baker Chemical Co. The preparation of dibenzamide has already been reported.[1] Dibenzoylmethane (Eastman Kodak Co.) was used as received.

Chloroform, *n*-hexane, and anhydrous diethyl ether were used as received from J. T. Baker Chemical Co. ("Baker

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[1] C. S. Kraihanzel and D. N. Stehly, *Inorg. Chem.*, **6**, 277 (1967).

[2] D. N. Stehly and C. S. Kraihanzel, presented in part at the 3rd Middle Atlantic Regional Meeting of the American Chemical Society, Philadelphia, Pa., Feb 1968.

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