

The Crystal Structure of 1-Ethyldecaborane

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Direct evidence for the existence of 1-ethyldecaborane has been established by a single-crystal X-ray structure analysis using, in part, the method of direct phase determination developed by Karle & Hauptman. The compound crystallizes in the orthorhombic system with $a = 10.11 \pm 0.01$, $b = 14.40 \pm 0.01$, and $c = 7.28 \pm 0.01$ Å. The space group is $P2_12_12_1$ and the unit cell contains four molecules of $B_{10}H_{13}C_2H_5$. The compound is a simple substitution derivative of decaborane and no significant distortion of the decaborane molecule is induced by the substitution.

Introduction

In the course of studies on decaborane derivatives, a compound was isolated which had a melting point near 10 °C. It was expected to be a single isomer of either an ethyldecaborane or a dimethyldecaborane. A single-crystal X-ray structure analysis was undertaken to establish which compound and which isomer of that compound had been isolated.

Unit cell and space group

The unit-cell dimensions, obtained from high angle reflections on zero level precession photographs which were corrected for film shrinkage, were

$$a = 10.11 \pm 0.01, \quad b = 14.40 \pm 0.01, \\ c = 7.28 \pm 0.01 \text{ Å.}^*$$

The estimated errors are based on earlier measurements of known crystals with the same camera.

The observed orthorhombic symmetry and the systematic extinctions $h00$, h odd; $0k0$, k odd; and $00l$, l odd established the space group unambiguously as $P2_12_12_1$. A comparison of the cell volume with the volumes occupied by similar compounds indicated 4 molecules of $B_{10}H_{13}C_2H_5$ per cell. Using the assumption of 4 molecules per cell the calculated density was 0.91 g.cm^{-3} .

Experimental

The compound $B_{10}H_{13}C_2H_5$ was supplied in sealed thin wall glass capillaries with a diameter of approximately 0.5 mm. A capillary was mounted on a precession camera and a single crystal was then grown by passing the capillary slowly through a stream of cold dry air. The apparatus allowed a single crystal to be preserved for several months while the data were being collected. The cold stream nozzle used was based on the design described by Post, Schwartz & Fankuchen (1951).

* All limits of error in this paper are to be considered as root-mean-square deviations.

The flow rate was regulated to maintain the crystal at approximately -20 °C.

Precession photographs using zirconium-filtered Mo radiation ($\lambda = 0.7107$ Å) were taken of the nkl ($n=0, 1, 2$), hnl ($n=0, 1, 2, 3$), $h\bar{h} + nl$ ($n=0, 1, 2, 3$), $2h, h, l$ and $h, 2\bar{h}, l$ layers. A series of timed exposures was made at each setting. Most of the intensities were measured on a densitometer and the very weak intensities were visually estimated. Of a possible 999 independent reflections, 296 were not observed. The intensities were corrected for differences in exposure times and corrected for Lorentz and polarization factors. No absorption corrections were considered necessary.

Structure determination

The treatment of the data and the sign determination procedures were carried out as outlined by Karle & Hauptman (1956) for the space group $P2_12_12_1$. The observed intensities, after correction for Lorentz and polarization factors, were corrected for vibrational motion and placed on an absolute scale by means of a K curve. From these absolute structure factor magnitudes the normalized structure factor magnitudes $|E_{hkl}|^2$ were computed from

$$|E_{hkl}|^2 = |F_{hkl}|^2 / \varepsilon \sum_{j=1}^N f_j^2(h, k, l)$$

where

$$\varepsilon(h00, 0k0, 00l) = 2 \\ \varepsilon = 1 \text{ for all other reflections.}$$

Initially all unobserved reflections were put in as zero intensity for the computation of the K curve and E values. However, when the average values of $|E|$ and $|E^2 - 1|$ from this set were computed, there was very poor agreement with the theoretical average values.

All of the unobserved reflections, except the space group extinctions, were then assigned an intensity equal to one-half the weakest observed intensity and the computation of the K curve and normalized

structure factors was repeated. The second K curve was indistinguishable from the first but the new set of $|E|^2$'s yielded averages in good agreement with the theoretical values.

The final values were

for 676 reflections (h, k, l all $\neq 0$, non-centrosymmetric)

	observed	theoretical
$\overline{ E ^2}$	1.014	1.000
$\overline{ E }$	0.871	0.886
$\overline{ E ^2 - 1 }$	0.834	0.736

for 322 reflections (h, k , or $l=0$, space group extinctions omitted, centrosymmetric)

	observed	theoretical
$\overline{ E ^2}$	1.038	1.000
$\overline{ E }$	0.807	0.798
$\overline{ E ^2 - 1 }$	1.038	0.968

The phase determining procedures were used to establish signs for the $hk0$ projection. The initial electron density projection was made with 51 of the stronger reflections out of a total of 85 observed $hk0$ data. Three of the 51 signs later proved to be wrong. This projection (Fig. 1) clearly showed the compound to be an ethyl, rather than a dimethyl, derivative and established the location of the boron framework in the unit cell. It was not clear enough to define the specific isomer unambiguously.

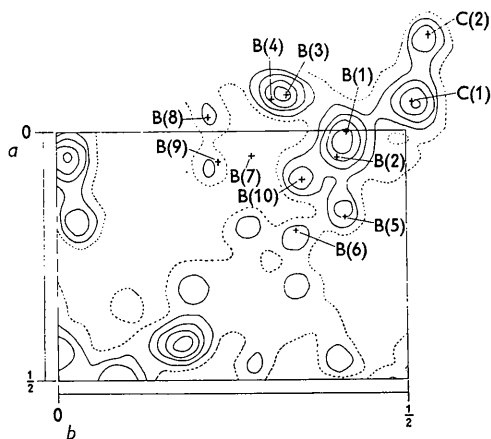


Fig. 1. First $hk0$ electron density projection using 51 reflections with all signs correct except the 220, 210, and 10,2,0. Final positions are indicated by +.

It was assumed that the boron atoms retained the decaborane configuration as determined by Kasper, Lucht & Harker (1950). Eleven possible orientations of this decaborane framework were found which could conceivably be consistent with the electron density projection. A least-squares refinement of the sets of x and y parameters for each model was attempted, the program of Busing & Levy (1959a) being used.

Nine of the models did not refine below $R=0.30$. One reduced to $R=0.26$ and one refined to $R=0.21$. In all of these cases a fixed isotropic temperature factor $B=3.5$ was used. The refinement of the most favorable case was continued with variable isotropic temperature factors and the R index reduced to a value of 0.17.

Examination of the structure factors resulting from the various models indicated that the signs of the 210 and 220 reflections must have been incorrect in the first electron density projection. Another electron density projection was made with the same 51 reflections, but with the 210 and 220 signs changed.

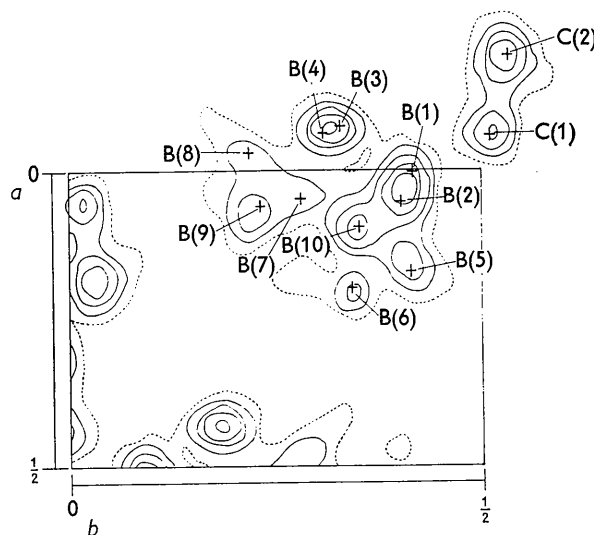


Fig. 2. Second $hk0$ electron density projection using the same 51 reflections as in Fig. 1, but with all signs correct except the 10,2,0. Final positions are indicated by +.

This projection, shown in Fig. 2, was much cleaner and in excellent agreement with the model which yielded the best refinement.

Approximate z parameters were obtained from the known molecular configuration in conjunction with the Harker section at $U = \frac{1}{2}$ of the Patterson synthesis. Improved z parameters were obtained by a least-squares refinement of the $0kl$ data. Further improvement of the position and individual isotropic temperature factor parameters for the boron and carbon atoms was obtained by several cycles of least squares using all of the observed data. The R value at this point was 16.1%.

At this stage a three-dimensional difference Fourier synthesis was computed in an attempt to locate the eighteen hydrogen atoms of the molecule. Twenty-one positive peaks of appreciable magnitude were observed in the asymmetric unit. The fifteen strongest of these were all very close to expected hydrogen positions. The remaining six peaks were of approximately equal magnitude, but only three were near possible hydrogen positions.

A final least-squares refinement was then run with 700 independent observed reflections. Three low order, very strong, reflections which were apparently affected by extinction were omitted. The x, y, z , and B parameters for the two C and ten B atoms, and the x, y, z , parameters for the eighteen H atoms were varied. The individual isotropic temperature factor of each H was arbitrarily fixed at 4.0. The final R from this last refinement was 0.095.

Attempts were made to vary the H temperature factors, but one or more of them always became negative. Inasmuch as all other features of the struc-

ture were quite satisfactory, this particular difficulty was not pursued further.

The atomic scattering factors used in this structure analysis were those of McWeeny (1951) for H, Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for C, and an average of the values given by James & Brindley (1931) and those of Nordman & Lipscomb (1953) for B.

Discussion of Karle-Hauptman procedure

The direct methods of structure determination are of great interest to crystallographers and some comments on the Karle-Hauptman procedure as used in this determination would seem warranted. The feasibility of the procedure in establishing phase magnitudes as well as signs for a non-centrosymmetric crystal is still untested in an actual structure determination. However, the present results do indicate that the procedure can be successfully used in establishing signs in centrosymmetric projections of non-centrosymmetric crystals.

In this work, efforts were concentrated on getting some sign indications for all the moderately strong $hk0$ reflections, *i.e.* with $|E| > 1.0$. Several signs were accepted on the basis of very weak sign indications that undoubtedly should not have been used on a more critical probability estimation. It is gratifying that, despite the uncritical use of weak sign determina-

Table 1. *Results of phase determination*

$hk0$	$ E $	Σ_1	Σ_3	Final Σ_2	Number of contributing pairs in Σ_2	Correct sign
0 16 0	2.657	-s	-w	-w	3	-
6 6 0	2.646	-w	+w	-s	9	-
8 0 0	2.527	-s	-s	-s	6	-
2 10 0	2.061	+s	+s	+s	7	+
10 10 0	1.953	-s	-s	-s	6	-
2 2 0	1.541	+m	+w	i	9	-
2 0 0	1.494	+w	+m	+m	9	+
0 12 0	1.391	+m	+m	+s	6	+
8 6 0	1.364	-w	-m	-m	7	-
6 2 0	1.184	+w	-m	+s	9	+
8 4 0	1.150	+w	+w	-s	8	-
4 6 0	1.075	-w	+m	-w	2	-
0 10 0	0.982	+m	-w	+m	11	+
10 2 0	0.972	-w	-s	-m	5	+
1 10 0	2.659	assumed	+	+s	17	+
7 4 0	2.278		-m	-s	13	-
1 2 0	1.905		-m	-s	22	-
5 8 0	1.619		-m	+m	14	+
9 8 0	1.346		-s	-m	13	-
1 6 0	1.304		-w	+m	11	+
3 4 0	1.147			-m	18	-
7 10 0	1.121			-s	11	-
5 10 0	1.065		-m	+w	12	+
7 2 0	0.929			+m	12	+
7 8 0	0.840			+m	11	+
1 8 0	0.842			+s	13	+
2 15 0	2.288	assumed	-	-s	7	-
4 7 0	2.068		+m	+w	10	+
10 5 0	1.986		+m	+m	4	+
10 7 0	1.676		-s	-m	4	-
2 5 0	1.361		-m	-m	5	-
2 9 0	1.281		-s	i	4	-
2 1 0	1.241		+w	+w	8	-
8 9 0	1.230		+s	+m	8	+
6 9 0	1.133		-m	-m	8	-
2 3 0	1.100		-s	-s	8	-
6 5 0	1.090		i	-m	5	-
7 7 0	2.219			+s	21	+
3 5 0	2.211			+s	27	+
11 3 0	2.052			+m	13	+
1 3 0	1.664			+s	30	+
1 13 0	1.659			-s	21	-
1 5 0	1.552			+s	25	+
7 9 0	1.433			-w	14	-
5 5 0	1.157			+s	26	+
7 5 0	1.123			+s	14	+
9 9 0	1.102			-w	12	-
9 7 0	1.007			+m	19	+
5 7 0	0.958			+m	18	+
7 3 0	0.924			+m	19	+
11 1 0	0.909			-s	15	-

Table 2. *Final atomic coordinates for C₂H₅B₁₀H₁₃ from least squares*

Positional parameter errors are ± 0.001 for B and C and ± 0.01 for H

Atom	x	y	z	B
C(1)	-0.057	0.510	0.011	4.0 \pm 0.2
C(2)	-0.191	0.533	0.104	5.0 \pm 0.2
B(1)	0.002	0.414	0.080	2.6 \pm 0.1
B(2)	-0.062	0.308	-0.018	2.3 \pm 0.1
B(3)	-0.072	0.330	0.224	2.7 \pm 0.2
B(4)	0.054	0.402	0.312	2.8 \pm 0.1
B(5)	0.097	0.351	-0.084	3.1 \pm 0.2
B(6)	0.061	0.231	-0.064	3.3 \pm 0.2
B(7)	-0.029	0.218	0.146	3.3 \pm 0.2
B(8)	0.049	0.281	0.364	3.1 \pm 0.2
B(9)	0.200	0.341	0.329	3.8 \pm 0.2
B(10)	0.172	0.413	0.132	3.4 \pm 0.2
H(2)	-0.15	0.32	-0.11	4.0
H(3)	-0.16	0.34	0.29	4.0
H(4)	0.04	0.46	0.41	4.0
H(5)	0.12	0.38	-0.23	4.0
H(6)	0.05	0.17	-0.16	4.0
H(7)	-0.10	0.15	0.16	4.0
H(8)	0.01	0.26	0.48	4.0
H(9)	0.28	0.35	0.43	4.0
H(10)	0.24	0.47	0.10	4.0
H(6-7)	0.08	0.19	0.09	4.0
H(5-6)	0.18	0.28	-0.07	4.0
H(9-10)	0.24	0.34	0.18	4.0
H(8-9)	0.15	0.25	0.32	4.0
H(C11)	0.02	0.57	0.03	4.0
H(C12)	-0.06	0.51	-0.14	4.0
H(C21)	-0.18	0.53	0.25	4.0
H(C22)	-0.22	0.61	0.05	4.0
H(C23)	-0.25	0.49	0.09	4.0

tions, sufficient correct signs were obtained to establish the basic structure with relative ease. The final criterion for determining a sign for the initial Fourier synthesis was the indication given by the Σ_2 formula, despite the fact that, based on the magnitudes of the summations and the small number of contributors, the indications would not be considered statistically valid for many of the determinations. For two of the reflections (220, 290) the Σ_2 summation was completely indeterminate and the Σ_3 indication was used.

Table 1 illustrates the sign indications obtained from the various formulae:

$$\Sigma_1: s(E_{hko}) = s \sum_{l_j} (-1)^{l_j+h_j} (|E_{h_j k_j l_j}|^2 - 1)$$

$$h = 2h_j$$

$$k = 2k_j$$

$$\Sigma_3: s(E_{hko}) = s \sum_{i,j} t^{\frac{1}{2}} E_i (|E_j|^2 - 1)$$

$$h = h_i + 2h_j, k = k_i + 2k_j$$

$$t = \frac{1}{2} \text{ if } l_j = 0$$

$$t = 1 \text{ if } l_j \neq 0$$

$$\text{or } h = h_i + 2h_j, k_i = 0, k = 2k_j$$

$$t = \sqrt{2}/2 \text{ if } l_j = 0$$

$$t = \sqrt{2} \text{ if } l_j \neq 0$$

$$\Sigma_2: s(E_{hko}) = s \sum E_i E_j$$

$$h = h_i + h_j, k = k_i + k_j, l = l_i + l_j.$$

A qualitative estimate of the magnitude of the summation is given as $s = \text{strong}$, $m = \text{medium}$, $w = \text{weak}$, $i = \text{indeterminate}$.

On the basis of the experience obtained from solving this structure, together with other work in this laboratory, there seems to be little doubt that the Σ_2 formula is the most powerful sign-determining relationship and can be successfully used with rather limited data.

Results and discussion

The final least-squares results for the atomic parameters of one molecule are given in Table 2. The numbering scheme used for the boron, carbon, and ethyl hydrogens is shown in Fig. 3(a). In the Tables, each hydrogen atom bonded to the decaborane molecule bears the number of the boron atom (or atoms in the case of bridge hydrogen) to which it is bonded.

The complete list of interatomic distances and standard errors, as computed by the Busing & Levy Function and Error Program (1959b), is given in Table 3. The corresponding distances for $B_{10}H_{14}$ (Moore, Dickerson & Lipscomb, 1957) are also given for comparison. In general, the agreement between the B-B distances of the two compounds is very close. The B(2)-B(6) and B(4)-B(9) distances appear to be significantly shorter than the other typical B-B distances in both compounds. The disagreement

between the B(1)-B(3) distances of the two compounds could be explained on simple steric considerations. In $B_{10}H_{13}C_2H_5$ the ethyl group is tucked under the decaborane framework (Fig. 3(b)) and the B(1), B(3), C(1) and C(2) atoms are very nearly coplanar. When all the hydrogen atoms in the vicinity are added the neighborhood becomes rather crowded.

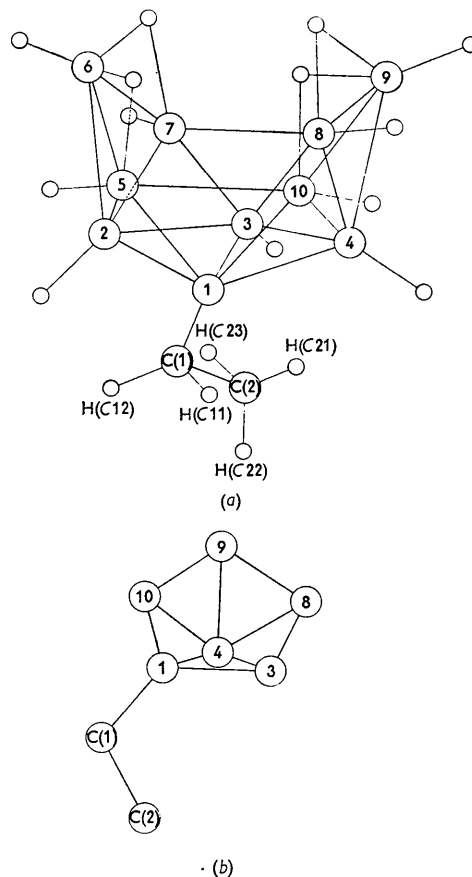


Fig. 3. (a) Molecular structure of $B_{10}H_{13}C_2H_5$ indicating the B, C, and ethyl H numbering used in this paper. (b) End view of molecule.

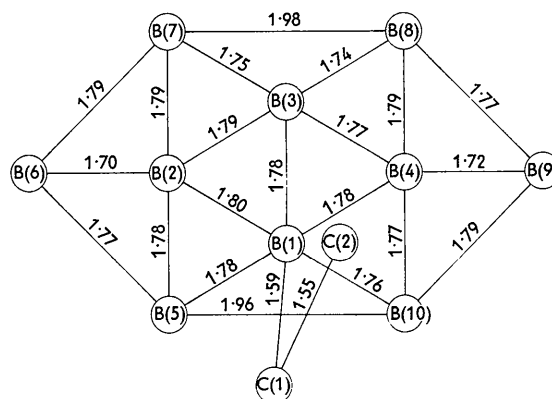


Fig. 4. B and C bond lengths (Å).

Table 3. *Interatomic distances*

	$C_2H_5B_{10}H_{13}$	$B_{10}H_{14}$		$C_2H_5B_{10}H_{13}$	$B_{10}H_{14}$
B(1)-C(1)	1.59 ± 0.01		B(6)-B(7)	1.79 ± 0.01	1.77
-B(2)	1.80 ± 0.01	1.80	-H(6)	1.11 ± 0.08	1.28
-B(3)	1.78 ± 0.01	1.71	-H(5-6)	1.36 ± 0.08	1.50
-B(4)	1.78 ± 0.01	1.78	-H(6-7)	1.31 ± 0.08	1.43
-B(5)	1.78 ± 0.01	1.78	B(7)-B(8)	1.98 ± 0.01	2.01
-B(10)	1.76 ± 0.01	1.77	-H(7)	1.23 ± 0.08	1.22
B(2)-B(3)	1.79 ± 0.01	1.78	-H(6-7)	1.28 ± 0.07	1.39
-B(5)	1.78 ± 0.01	1.76	B(8)-B(9)	1.77 ± 0.01	1.77
-B(6)	1.70 ± 0.01	1.72	-H(8)	0.97 ± 0.09	1.35
-B(7)	1.79 ± 0.01	1.80	-H(8-9)	1.13 ± 0.08	1.25
-H(2)	1.11 ± 0.08	1.27	B(9)-B(10)	1.79 ± 0.01	1.77
B(3)-B(4)	1.77 ± 0.01	1.80	-H(9)	1.12 ± 0.08	1.28
-B(7)	1.75 ± 0.01	1.77	-H(8-9)	1.34 ± 0.07	1.50
-B(8)	1.74 ± 0.01	1.78	-H(9-10)	1.18 ± 0.09	1.43
-H(3)	1.00 ± 0.07	1.16	B(10)-H(10)	1.13 ± 0.08	1.22
B(4)-B(8)	1.79 ± 0.01	1.76	-H(9-10)	1.25 ± 0.08	1.39
-B(9)	1.72 ± 0.01	1.72	C(1)-C(2)	1.55 ± 0.01	
-B(10)	1.77 ± 0.01	1.80	-H(C11)	1.16 ± 0.07	
-H(4)	1.15 ± 0.08	1.27	-H(C12)	1.10 ± 0.09	
B(5)-B(6)	1.77 ± 0.01	1.77	C(2)-H(C21)	1.08 ± 0.08	
-B(10)	1.96 ± 0.01	2.01	-H(C22)	1.20 ± 0.08	
-H(5)	1.14 ± 0.08	1.35	-H(C23)	0.91 ± 0.09	
-H(5-6)	1.30 ± 0.08	1.25			

The stretching of the B(1)-B(3) distance to the observed magnitude could be ascribed to the crowding.

Although there are no constraints placed on the molecular symmetry by the crystal lattice, it can be

seen from Fig. 4 and Table 3 that the decaborane framework retains, within the limits of error, the $mm2$ (C_{2v}) symmetry of the unsubstituted decaborane molecule. The symmetry of the ethyldecaborane is

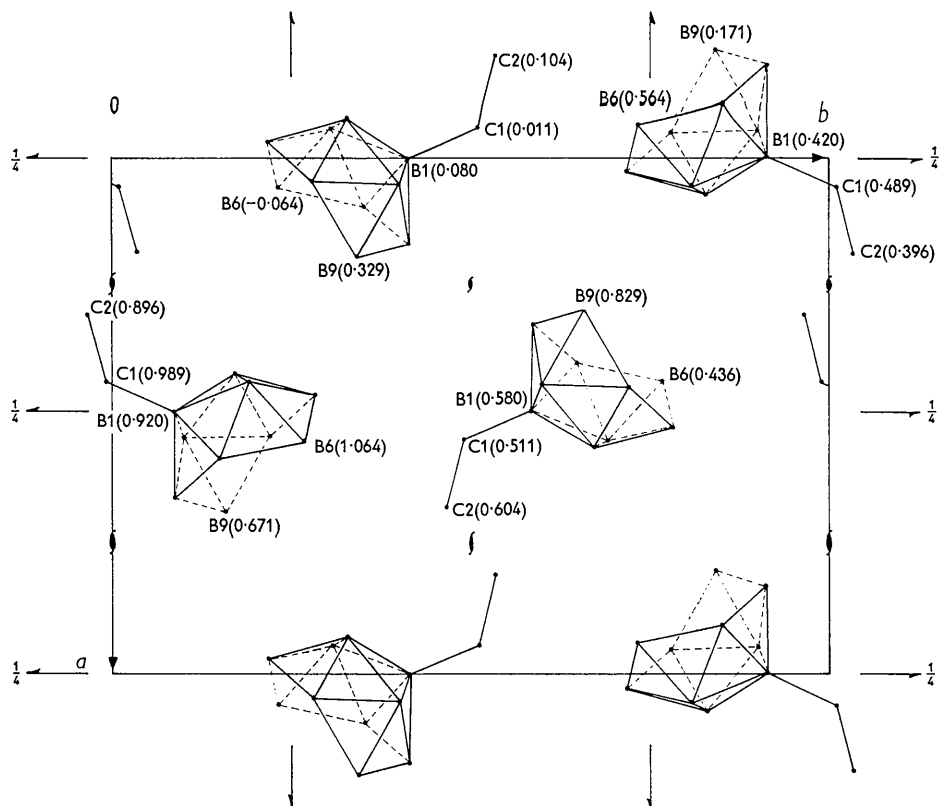


Fig. 5. Molecular packing. The upper part of the molecule is linked by solid lines and the lower part by dashed lines. The z parameters of B(1), B(6), B(9), C(1), and C(2) are given to illustrate relative heights of molecules.

Table 4. Observed and scaled calculated structure factors
The scale factor (s) is 2.19. Reflections marked * were omitted from the final least-squares refinement

Table with 5 columns of reflection data. Each column lists reflections (hkl) and their corresponding structure factor values (Fo, s|Fol, 10Ac, 10Bc). Reflections are grouped by their Miller indices.

very close to m (Cs). If we consider the plane defined by B(1), B(3), and the midpoint between B(6) and B(9) as the approximate mirror plane, then C(1) is 0.01 Å and C(2) is 0.13 Å from the plane. The deviations from m (Cs) symmetry can be ascribed to the packing requirements in the crystalline state.

The arrangement of C2H5B10H13 molecules in the unit cell is illustrated in Fig. 5.

The distances involving hydrogen atoms are not very accurate, but, in general, they agree reasonably with the values reported for other boron hydrides. The average normal B-H distance for B10H13C2H5

is 1.11 Å and the average bridge B–H distance is 1.27 Å. These results are not accurate enough to confirm or refute the asymmetry of the bridge hydrogen atoms.

The present results establish that simple substitutional derivatives of decaborane are formed which retain the essential symmetry of the isolated decaborane molecule, and confirm the existence of a 1-ethyldecaborane which has been suggested by Blay, Dunstan & Williams (1960) on the basis of chemical and nuclear magnetic resonance evidence.

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The Relative Positions of Independent Molecules Within the Same Asymmetric Unit

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If \mathbf{x} and \mathbf{x}' are position vectors of equivalent points in identical molecules in different parts of the crystallographic asymmetric unit, then the linear relationship between these points may be written as $\mathbf{x}' = [\mathbf{C}]\mathbf{x} + \mathbf{d}$, when $[\mathbf{C}]$ is a rotation matrix and \mathbf{d} a translation vector. A function is derived for determining \mathbf{d} , given $[\mathbf{C}]$, when no knowledge of the phases is available.

Consider a crystal structure which contains two or more molecules, or other identical distributions of electron density, within the crystallographic asymmetric unit. Such distributions of density will be referred to as sub-units. In a previous paper we have shown how the angular relationship between the sub-units may be derived from the Patterson function (Rossmann & Blow, 1962). This depended on the idea that the Patterson vectors within one sub-unit (the 'self-vectors') formed a similar distribution for each sub-unit; so that a rotation can be found which brings the self-vectors from one sub-unit into coincidence with those from the other.

In this paper we shall consider how the 'cross-vectors', or Patterson vectors from one sub-unit to another, can be used to determine the translation required to bring one sub-unit into coincidence with the other, after suitable rotation about a given axis. In other papers (Hodgkin, Harding, Coller & Rossmann, 1964; Blow, Rossmann & Jeffery, 1964) we describe the application of this method to the proteins insulin and chymotrypsin. In a further paper (Rossmann & Blow, 1963) we have suggested how the condition that the sub-units must be identical, once their relative orientation and position are known, may be used to determine phases, thus leading to a solution of the structure.

Although the result of a rotation and translation in three dimensions depends on the position of the

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