Å, c = 7.967 (5) Å, and β = 93° 47 (1)′, with least-squares standard errors in parentheses. An approximate correction for the ^{249}Cf content of the sample was made by assuming a linear change in all lattice dimensions with ionic radius, proceeding from Am to Cf; the slightly larger, corrected dimensions of the $BkCl_3 \cdot 6H_2O$ cell are a = 9.658 Å, b = 6.536 Å, c = 7.969 Å, and β = 93° 46′. The indexed powder pattern, calculated from this cell, is listed along with the observed lines in Table IV. That there is only rough qualitative agreement between observed and calculated intensities is probably due to the preferred orientation and limited number of crystals in the powder sample.

Other Hydrates.—Our values for the dimensions of the reduced[23] triclinic unit cells of the trichloride heptahydrates of La, Ce, and Pr are listed in Table V along with the reduced[23] cells derived from the cells of Iveronova, *et al.*,[7] for comparison. The reduced cells should be in agreement, but are not. We believe that there is an error in the published values of Iveronova, *et al.*, because their cell volumes are too small when compared with the expected values obtained by extrapolation of the hexahydrate cell volumes plus the volume of one additional H_2O per molecule.

Since the trichlorides of these three largest lanthanide ions form heptahydrates and the trichlorides of Nd–Lu form hexahydrates, it is of interest to compare the actinide trichloride compounds with corresponding radii. With respect to size of the trivalent ion, $AmCl_3 \cdot 6H_2O$ and $BkCl_3 \cdot 6H_2O$ are bracketed by the known $NdCl_3 \cdot 6H_2O$ and $EuCl_3 \cdot 6H_2O$. Using only ionic size as a criterion, one would expect that if uranium and neptunium trichlorides form stable hydrates they should contain seven water molecules, whereas all the trichlorides of actinides heavier than Pu should form hexahydrates. Nevertheless, tribromide hexahydrates with the structure discussed here are known for U, Np, and Pu, but the slightly larger Br ions may be effective in preventing a seventh water molecule from entering the complex cation.

(23) The reduction was carried out by the method of Delaunay as described in the "International Tables for X-Ray Crystallography," Vol. I, Kynoch Press, Birmingham, England, 1952, p 530.

TABLE V

TRICLINIC UNIT-CELL DIMENSIONS[a] OF LANTHANIDE TRICHLORIDE HEPTAHYDRATES

Compound	a, Å	b, Å	c, Å	α, deg	β, deg	γ, deg	V, Å^3
$LaCl_3 \cdot 7H_2O$	10.44	8.18	8.03	91.8	104.1	122.1	552.6
$LaCl_3 \cdot 7H_2O$	(10.1	8.1	10.2	98.8	117.9	121.4	534)[b]
$CeCl_3 \cdot 7H_2O$	10.43	8.20	7.98	91.9	104.0	122.1	549.6
$PrCl_3 \cdot 7H_2O$	10.29	8.22	7.90	91.9	105.1	121.3	540.3
$PrCl_3 \cdot 7H_2O$	(10.1	8.2	10.1	98.9	117.2	122.3	536)[b]

[a] Our values have an estimated error of ±0.5%. [b] Values in parentheses are derived from Iveronova, *et al.*[7]

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Crystal Structure of Bis(triethylammonium) Octadecahydroicosaborate. The Structure of Octadecahydroicosaborate(2−)

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The centrosymmetric $B_{20}H_{18}^{2-}$ ion, octadecahydroicosaborate(2−), consists of two B_{10} units, very similar to that in $B_{10}H_{10}^{2-}$, linked by B···B interaction among one pair of adjacent apical and equatorial B atoms from each B_{10} unit. These interactions are interpreted as involving two localized three-center BBB bonds among these four B atoms. Location of all H atoms indicates that no hydrogen bridges are present. The triethylammonium ion has approximately C_3 symmetry. Twofold positional disorder exists in all ions in the monoclinic crystal, which has symmetry $P2_1/a$, and two formula weights per unit cell having parameters a = 17.52 (2), b = 7.60 (1), c = 11.72 (1) Å, and β = 109.7 (2)°. The value of $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ is 0.10 for the 1874 X-ray diffraction maxima.

The polyhedral $B_{10}H_{10}^{2-}$ ion, first prepared and recognized by Lipscomb, Pitochelli, and Hawthorne,[1] was proposed[2] to dimerize by formation of two hydrogen bridges to form the $B_{20}H_{18}^{2-}$. Subsequently, $B_{20}H_{18}^{2-}$ was prepared,[3] but the nuclear magnetic resonance study by Pilling, Hawthorne, and Pier[4] indicated linkage of the B_{10} units by two three-center BBB bonds rather than through bridge hydrogens. The boron framework was confirmed in Babcock's X-ray study[5] of $[(CH_3)_3S]_2$-$B_{20}H_{18}$, but hydrogen atoms were not located and the B–B distances have not been published. Thus, the possibility of additional bonding through unsymmetri-

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(1) W. N. Lipscomb, A. R. Pitochelli, and M. F. Hawthorne, *J. Amer. Chem. Soc.*, **81**, 5833 (1959).

(2) W. N. Lipscomb, *Proc. Nat. Acad. Sci. U. S.*, **47**, 1791 (1961).

(3) A. Kaczmarczyk, R. D. Dobrott, and W. N. Lipscomb, *ibid.*, **48**, 729 (1962).

(4) R. L. Pilling, M. F. Hawthorne, and E. A. Pier, *J. Amer. Chem. Soc.*, **86**, 3568 (1964).

(5) B. L. Chamberland and E. L. Muetterties, *Inorg. Chem.*, **3**, 1450 (1964).

cal hydrogen bridges remains, especially since the recent preparation[6] and X-ray study[7] of a photoisomer of $B_{20}H_{18}^{2-}$ does indeed show two B_{10} units joined by hydrogen bridges. However, we shall show that no hydrogen bridges exist in the normal isomer in the X-ray diffraction study described below, where all hydrogen atoms were located.

Experimental Section

Colorless, lath-shaped crystals were grown from water–methanol solution. The crystals are monoclinic, and unit cell parameters are a = 17.52 (2), b = 7.60 (1), c = 11.72 (1) Å, and β = 109.7 (2)°, where parentheses indicate the estimated errors in the last place. These parameters were determined from an $hk0$ Weissenberg film which had been calibrated with an external standard (cubic, a = 4.0415 Å) and from scattering angles measured on the Buerger automated diffractometer. Their estimated errors are calculated standard deviations multiplied by five, and these parameters were measured with the use of Cu Kα (λ 1.5418 Å) radiation and at a crystal temperature of 22 (2)°. Assumption of two formula weights per unit cell gives a calculated density of 0.991 g cm^{-3} in agreement with the value of 0.982 (10) g cm^{-3} as determined by flotation in *m*-xylene–bromobenzene solution. Weissenberg films of levels $h0l$, $hk0$, and $hk3$, taken with Cu Kα radiation, showed absences for $h0l$ when h is odd and of $0k0$ when k is odd. Thus, the probable space group is $P2_1/a$, and each $B_{20}H_{18}^{2-}$ ion must lie on a center of symmetry or be disordered about this symmetry center.

Intensities were measured on a Supper-Pace automated Buerger diffractometer, with the use of Ni-filtered Cu Kα radiation and a scintillation counter. This instrument employs Weissenberg geometry and an ω scan with a (horizontal) spindle coordinate ϕ incremented through 2 + kL degrees at 1°/min, where L is the Lorentz factor and k is a constant (0.4–0.7). Stationary background counts, taken for 45 sec before and after the scan, were summed, corrected to correspond to the scan time, and then subtracted from the scan count to give the uncorrected intensity. Only positive intensities were retained. Empirical errors, evaluated from a different study, were assigned as 0.02 times the uncorrected intensity plus the square root of the sum of scan count and background. These error estimates tend toward a proportion of the intensity for large intensities, and toward counter statistics for smaller intensities.

The crystal was 8 cm from the source and 16 cm from the detector. A 1.0- or 1.2-mm collimator was employed for the incident beam, which was at a takeoff angle of 6°, and the diffracted beam passed through three apertures of minimum size 2.2 mm located 12–15 cm from the crystal. Pulses from the counter were recorded if simultaneously passed by a pulse height analyzer and a pulse shape discriminator. After every tenth reflection a check reflection was measured, and after every level six zero-level reflections were measured. We found that after about 3 weeks of irradiation the most sensitive check reflection dropped by 10%, and at that time discarded the crystal. No correction was required for coincident losses, inasmuch as a linear relation was found for the log of the count rate *vs.* thickness of an attenuating foil for the most intense reflection. Data were taken on three crystals of dimensions 0.9 × 0.5 × 0.3, 0.9 × 0.7 × 0.3, and 0.5 × 0.3 × 0.2 mm, rotated about the longest axis, [1$\bar{1}$0], [010], and [100], respectively. The last of these crystals was previously dipped into liquid N_2 in order to reduce extinction, for which no corrections were made. These data included levels 0 through 11 about [1$\bar{1}$0], levels 0 through 4 about b, and all accessible intense reflections about a. The absorption coefficient μ = 3.19 cm^{-1} gave minimum and maximum corrections to F_{hkl}'s of 1.05–1.08, 1.05–1.12, and 1.03–1.05 for these three crystals, respectively, on the zero level, and of 1.06–1.10, 1.06–1.14, and 1.04–1.06 at an equiinclination angle of 30°. Since each level for each crystal was given its own scale factor during correlation, no further absorption corrections were made. After application of Lorentz and polarization corrections, the data were scaled and averaged to yield a value of $R = \Sigma_{H,i}|S_iI_{Hi} - \bar{I}_H|/\Sigma_{H,i}\bar{I}_H$ equal to 0.04, where H is hkl, S_i is the scale factor of level i, I_{Hi} is the intensity of reflection H on level i, and $\bar{I}_H$ is the average value of reflection H. An initial estimate, by Wilson's method, of the scale factor was refined later by least-squares procedures. Observed and calculated structure factors (×10) are given in Table I, where those reflections not included in the refinement are marked with an asterisk.

Structure Determination and Refinement

The structure was solved by reiterative application of Sayre's equation.[8] First, normalized structure factors E_H were calculated for the [1$\bar{1}$0] axis data (correlated with 60 roughly measured $hk0$ and $hk1$ reflections) from the equation[9]

$$E_H^2 = F_H^2/\left(\epsilon \sum_{j=1}^{N} f_{jH}^2\right)$$

in which F_H^2 has been corrected for thermal motion, the f_{jH} are atomic scattering factors, N is the number of atoms per unit cell, and ϵ is a multiplicity factor equal to 2 for $h0l$ and $0k0$ reflections and 1 otherwise. A modified version of the computer program of Long[10] was used to determine signs for those 316 reflections having $E \geq 1.30$. These reflections were ordered in decreasing size of $|E_H|\Sigma_K|E_K||E_{H-K}|$. The first three that were linearly independent were fixed in sign in order to determine the origin,[11] and the first four remaining were arbitrarily given signs. For each of the resultant 16 sets of assumed signs, Sayre's equation was then repeatedly applied until a self-consistent set of signs appeared or until 17 cycles elapsed. These E values were weighted by $w_H = |2P_H^+ - 1|$, where P_H^+ is the probability that reflection H has a positive sign. The correct solution converged in the fewest cycles and showed the highest weighted consistency index

$$C = \frac{\langle|w_HE_H\sum_K w_KE_Kw_{H-K}E_{H-K}|\rangle}{\langle|E_H|\sum_K|E_K||E_{H-K}\rangle} = 0.66$$

independent of procedures in which newly determined signs were used immediately or in which these signs were used only in the following cycle. Only two signs of this set changed during later refinement of the structure.

A stereoview of the $B_{20}H_{18}^{2-}$ ion is shown in Figure 1 and the atomic numbering scheme is shown in Figures 2–5, where two orientations of the B_{10} cage in the disorder have apices labeled as 1,10 and 13,20. The map made from the 316 E's revealed borons 11, 2, 3, 4, 5, 16, 7, 18, 9, 10, and 20 at heights of 251–659 arbitrary units, nitrogen 21 at 681, carbons 22, 23, 24, 28, 29, and 30 at 346–441, and the highest false peak at 230. The borons formed a plausible cage, but one could incorporate

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TABLE I

OBSERVED AND CALCULATED STRUCTURE FACTORS[a] (×10) FOR $[(C_2H_5)_3NH]_2B_{20}H_{18}$

[a] Columns contain h index, F_o, and F_c. Asterisks denote reflections not used in refinement; dashes, those with zero or negative measured intensities.

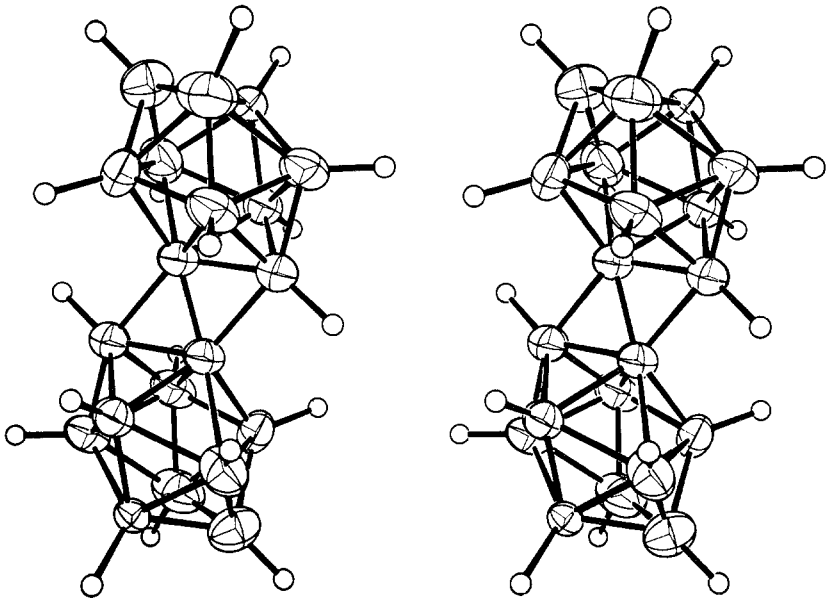

Figure 1.—Stereoview of the $B_{20}H_{18}^{2-}$ ion in the 1,10 apex form.

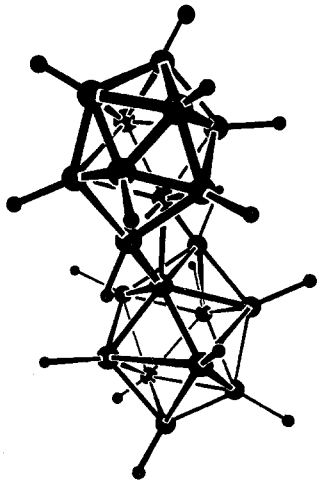

Figure 2.—The $B_{20}H_{18}^{2-}$ ion in the 1,10 apex form. Direction cosines of the projection axis with respect to the crystallographic axes are 0.20, 0.78, 0.50.

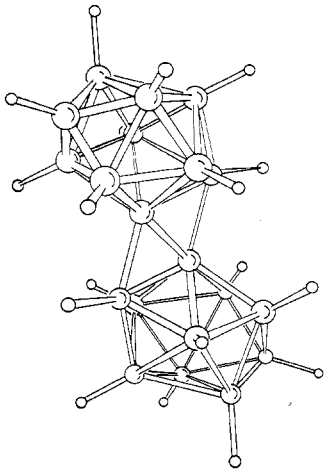

Figure 3.—The $B_{20}H_{18}^{2-}$ ion in the 13,20 apex form, projected as in Figure 2.

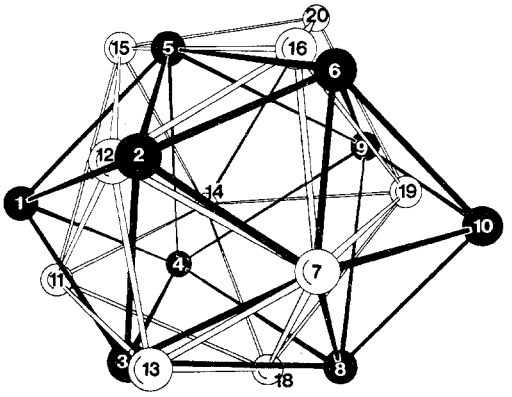

Figure 4.—Superposition of the 1,10 and 13,20 forms showing the disorder. Direction cosines of the projection axis with respect to the crystallographic axes are −0.49, 0.29, 0.94.

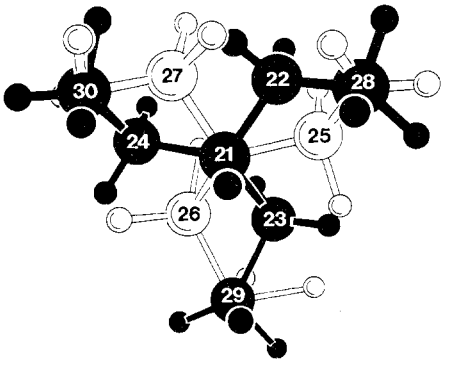

Figure 5.—The triethylammonium ion and its disorder. Direction cosines of the projection axis with respect to the crystallographic axes are 0.70, 0.10, 0.42.

either B(10) or B(20) at one apex. We chose to omit B(20) and computed structure factors which yielded $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.42$. In these and all later computations, atomic scattering factors were taken from ref 12. Least-squares procedures were carried out in

(12) "International Tables for X-Ray Crystallography," Vol. 3, Kynoch Press, Birmingham, England, 1962, pp 202–203.

which we minimized $\Sigma w(|F_o|^2 - |F_c|^2)^2$ where $\sqrt{w}$ was taken as $1/\sigma(|F_o|^2)$, and $\sigma(|F_o|^2)$ was assumed to be proportional to $|F_o|$ for $|F_o| \geq 4.8$ and to $\sqrt{4.8|F_o|}$ for $|F_o| < 4.8$. At this stage (B(20) omitted) least-squares refinement of positions and isotropic temperature factors converged only to $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.33$, and the temperature factors for methylene carbons 22, 23, and 24 became greater than 10 Å^2. In an electron density map atom B(20) appeared, but no new peaks were found. The procedure was repeated with atom B(20) included, but B(10) omitted, but refinement was similarly unsuccessful.

After the usual interval, it became clear that both B(10) and B(20) could be included either in a disordered structure in the space group $P2_1/a$ or in an ordered structure in space group Pa having all odd $0k0$ reflections accidentally absent. This latter possibility, involving a twisted, noncentrosymmetric $B_{20}H_{18}^{2-}$ ion, was rejected when a structure factor calculation yielded fairly large values for several odd $0k0$ reflections.

The $[1\bar{1}0]$ and b axis data were correlated and introduced at this point. Repeated calculations consisting of (1) examination of a Fourier or difference Fourier map for new peaks, (2) introduction of atoms for which the disorder was resolved with a population of 0.5 at each site, and (3) two cycles of isotropic refinement for all atoms, reduced R in stages to 0.29, 0.28, and 0.23, and finally located all nonhydrogen atoms. In Table II we show the separation between disordered atoms, the stage at which the disorder was resolved, and the height at which the second component appeared. Atom B(7) is not disordered, and B(4) and B(9) appeared on the E map almost halfway toward B(14) and B(19). The isotropic temperature factors of methylene carbons 25, 26, and 27 were much higher than those for 22, 23, and 24. Adjustment of the populations of those two groups of carbon atoms to 0.4 and 0.6, respectively, allowed these temperature factors to equalize upon further refinement.

TABLE II
RESOLUTION OF DISORDERED ATOMS[a]

Pair	R factor	Height, e$^-$/Å^3	Sepn, Å
11,1	23	0.7	0.71
2,12	23	1.0	0.40
3,13	29	1.7	0.54
4,14	23	1.0	0.67
5,15	23	0.6	0.46
16,6	23	0.8	0.46
18,8	29	1.6	0.63
9,19	23	0.9	0.57
10,20	E map		2.65
22,25	28	0.7	1.19
23,26	28	0.7	1.37
24,27	28	1.00	1.30

[a] Given are the R factor at which the second component appeared on the map, height at appearance, and distance between components.

At this point the a axis data were introduced, a final isotropic refinement lowered R to 0.205, and a difference electron density map revealed all hydrogen atoms of the boron cages. Hydrogens 31–50, attached to borons 1–20, generally appeared as ellipsoids of maximum

TABLE III
POSITIONS AND ANISOTROPIC TEMPERATURE FACTORS[a] WITH STANDARD DEVIATIONS FOR B, C, AND N ATOMS

Atom	Pop.	x	y	z	$10^4\beta_{11}$	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{13}$	$10^4\beta_{23}$
B(1)	0.5	0.3956 (5)	0.2191 (13)	0.2084 (7)	41 (5)	245 (22)	54 (7)	39 (15)	31 (9)	36 (20)
B(2)	0.5	0.3701 (7)	−0.0210 (11)	0.1940 (9)	46 (6)	156 (19)	72 (11)	−17 (14)	35 (12)	−19 (20)
B(3)	0.5	0.4667 (6)	0.0915 (11)	0.1894 (7)	45 (5)	149 (21)	66 (8)	13 (13)	65 (9)	7 (20)
B(4)	0.5	0.4806 (5)	0.2193 (12)	0.3273 (7)	46 (5)	204 (21)	71 (10)	−33 (16)	56 (11)	−43 (22)
B(5)	0.5	0.3769 (6)	0.1254 (11)	0.3274 (9)	32 (5)	179 (20)	93 (10)	22 (14)	56 (11)	−25 (22)
B(6)	0.5	0.3976 (5)	−0.1040 (11)	0.3435 (8)	29 (4)	170 (18)	51 (8)	−5 (13)	13 (9)	−28 (18)
B(7)	1.0	0.4605 (3)	−0.1199 (6)	0.2654 (4)	34 (2)	233 (10)	99 (4)	−11 (7)	21 (5)	9 (11)
B(8)	0.5	0.5373 (5)	0.0224 (13)	0.3353 (8)	32 (4)	251 (28)	85 (9)	7 (16)	45 (10)	−81 (26)
B(9)	0.5	0.4742 (5)	0.0539 (10)	0.4337 (7)	27 (4)	144 (18)	54 (7)	−3 (14)	23 (8)	14 (17)
B(10)	0.5	0.5031 (5)	−0.1483 (11)	0.4061 (6)	37 (4)	179 (20)	57 (7)	2 (13)	30 (8)	−46 (18)
B(11)	0.5	0.4378 (6)	0.2192 (16)	0.2183 (8)	47 (7)	321 (32)	90 (11)	−44 (23)	16 (13)	100 (30)
B(12)	0.5	0.3735 (5)	0.0312 (8)	0.1982 (7)	22 (4)	89 (14)	49 (8)	−4 (9)	14 (9)	−15 (14)
B(13)	0.5	0.4628 (7)	0.0248 (12)	0.1717 (8)	57 (6)	169 (25)	90 (10)	5 (16)	56 (12)	−49 (24)
B(14)	0.5	0.4749 (5)	0.2419 (12)	0.3790 (7)	39 (4)	186 (21)	72 (9)	14 (15)	0 (9)	−29 (22)
B(15)	0.5	0.3703 (6)	0.1819 (11)	0.3148 (8)	36 (5)	184 (21)	65 (9)	0 (14)	5 (10)	−24 (21)
B(16)	0.5	0.3870 (6)	−0.0489 (10)	0.3442 (8)	28 (4)	168 (19)	61 (9)	−6 (13)	22 (9)	17 (17)
B(18)	0.5	0.5262 (5)	0.0967 (12)	0.3114 (7)	33 (4)	182 (22)	84 (8)	−13 (14)	57 (9)	−39 (22)
B(19)	0.5	0.4938 (5)	−0.0015 (12)	0.4269 (7)	29 (4)	205 (22)	56 (8)	0 (16)	21 (9)	29 (22)
B(20)	0.5	0.4216 (5)	0.1152 (11)	0.4562 (6)	33 (4)	193 (18)	61 (7)	28 (14)	27 (8)	14 (19)
N(21)	1.0	0.3235 (2)	0.4121 (4)	0.7792 (2)	38 (1)	169 (6)	63 (3)	8 (5)	24 (3)	3 (7)
C(22)	0.6	0.3359 (4)	0.3691 (10)	0.6621 (5)	55 (3)	300 (18)	76 (6)	102 (13)	68 (7)	44 (17)
C(23)	0.6	0.3748 (5)	0.5774 (10)	0.8307 (6)	82 (5)	236 (19)	107 (8)	−82 (16)	19 (10)	−66 (21)
C(24)	0.6	0.3449 (4)	0.2594 (9)	0.8680 (5)	62 (4)	208 (15)	67 (6)	38 (12)	27 (7)	59 (15)
C(25)	0.4	0.3595 (7)	0.5147 (17)	0.6935 (9)	54 (6)	373 (32)	104 (10)	−59 (22)	70 (12)	84 (32)
C(26)	0.4	0.3737 (8)	0.4446 (16)	0.9092 (9)	84 (8)	342 (37)	104 (13)	−67 (28)	25 (16)	−196 (35)
C(27)	0.4	0.3268 (7)	0.2105 (13)	0.7557 (11)	75 (7)	154 (25)	170 (15)	27 (21)	81 (17)	0 (33)
C(28)	1.0	0.3045 (3)	0.5025 (6)	0.5635 (3)	53 (2)	306 (11)	90 (4)	27 (7)	60 (4)	85 (11)
C(29)	1.0	0.3646 (3)	0.6421 (7)	0.9420 (4)	89 (3)	327 (14)	143 (6)	−32 (11)	59 (7)	−114 (15)
C(30)	1.0	0.2863 (3)	0.1078 (6)	0.8281 (4)	73 (3)	214 (11)	140 (5)	−7 (9)	62 (6)	72 (13)

[a] The x, y, and z are in fractional monoclinic coordinates. Temperature coefficients occur in the expression $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + kl\beta_{23})]$.

height 0.24–0.45 $e^-/Å^3$ from which the two disordered components had to be approximated. Fortunately, however, the two hydrogen atoms of greatest interest to us were clearly resolved, as H(40) and H(50), bonded, respectively, to B(10) and B(20). These were the hydrogens which distinguish whether there are hydrogen bridges or terminal hydrogens (or some intermediate structure) between the two B_{10} cages of $B_{20}H_{18}^{2-}$. Hydrogens 51–78 in the triethylammonium group varied from well-resolved peaks of height 0.38–0.48 $e^-/Å^3$ for ammonium hydrogen 51 and methyl hydrogens 64, 69, and 74 (which are at population 1.0 and are common to both forms of the disorder) to barely discernible bulges for some hydrogens with population 0.4.

Inclusion of H atoms in fixed positions and refinement alternatively of coordinates and anisotropic temperature factors for N, C, and B atoms yielded $R = 0.106$. As a study of the disorder of methylene carbons, least-squares refinements were made with populations either of 0.6 and 0.4 or of 0.5 and 0.5, and a plot was made of the largest, middle, and smallest principal axis values of the anisotropic temperature factors of these disordered atoms *vs.* deviation of the population either way from 0.5 and 0.5. The results indicated the distribution 0.62–0.38. At this stage, those hydrogen atoms lying on gradients on a difference Fourier were adjusted, and, finally, the positions of the hydrogen atoms 40 and 50 were refined. The final R factor was 0.103 for 1874 reflections, the weighted R factor $R_w = [\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w|F_o|^4]^{1/2}$ was 0.194, and the standard deviation of an observation of unit weight was 1.68. A final difference electron density map had two sizable peaks of height 0.31 and 0.22 $e^-/Å^3$, roughly the height of 0.6 hydrogen or 0.2 carbon. The larger peak occurs very near the included hydrogen atom 70 and may be the result of a disorder in methyl carbon 29. All other peaks are less than 0.15 $e^-/Å^3$ in height.

Results and Discussion

Positions and temperature factors are given in Table III for B, C, and N atoms and in Table IV for H atoms. For those atoms refined anisotropically the root-mean-square amplitudes of vibration and direction cosines of the thermal ellipsoids relative to the crystallographic axes are listed in Table V. Bond distances are provided in Table VI; bond angles, in Table VII.

The $B_{20}H_{18}^{2-}$ ion (Figure 1) closely fits a model of C_{2h} symmetry, in which the plane of symmetry passes through atoms 9, 10, 9′, 10′ or 19, 20, 19′, 20′. Boron atoms deviate at most by 0.1 Å from the symmetry-required positions for C_{2h}, and hydrogen atoms with one exception deviate at most by 0.2 Å. The B_{10} cage seems little changed from its structure in $Cu_2B_{10}H_{10}$.[13] The degree of anisotropy in the thermal motion is not large, and the values of the thermal parameters suggest that the thermal motion is relatively undisturbed by the disorder. Average apex-to-edge boron cage distances with standard deviations are 1.71 (9) Å for the 1,10 orientation and 1.70 (7) Å for 13,20 compared with 1.73 (2) Å in $Cu_2B_{10}H_{10}$.[13] Corresponding edge–edge distances along the base of a square pyramid are 1.83 (10) and 1.85 (7) Å [1.86 (2) Å in $Cu_2B_{10}H_{10}$]. Along the equatorial belt they are 1.79 (5) and 1.84 (9) Å

TABLE IV
POSITIONS[a] AND ASSIGNED ISOTROPIC TEMPERATURE FACTORS[b] FOR H ATOMS

Atom	Pop.	x	y	z
		Attached to Boron		
31	0.5	0.350 (9)	0.313 (20)	0.160 (13)
32	0.5	0.310 (9)	−0.083 (20)	0.124 (13)
33	0.5	0.498 (9)	0.104 (20)	0.111 (13)
34	0.5	0.531 (9)	0.333 (20)	0.381 (13)
35	0.5	0.323 (9)	0.188 (20)	0.374 (13)
36	0.5	0.343 (9)	−0.187 (20)	0.367 (13)
37	0.5	0.470 (13)	−0.236 (30)	0.213 (19)
38	0.5	0.607 (9)	0.049 (20)	0.340 (13)
40	0.5	0.540 (5)	−0.273 (10)	0.436 (6)
41	0.5	0.425 (9)	0.361 (20)	0.154 (13)
42	0.5	0.313 (9)	−0.031 (20)	0.111 (13)
43	0.5	0.491 (9)	0.021 (20)	0.094 (13)
44	0.5	0.505 (9)	0.375 (20)	0.436 (13)
45	0.5	0.311 (9)	0.243 (20)	0.309 (13)
46	0.5	0.341 (9)	−0.146 (20)	0.361 (13)
47	0.5	0.461 (13)	−0.250 (30)	0.278 (19)
48	0.5	0.601 (9)	0.125 (20)	0.345 (13)
50	0.5	0.400 (5)	0.190 (10)	0.535 (6)
		Attached to Nitrogen		
51	1.0	0.258 (5)	0.458 (10)	0.755 (6)
		Attached to Methylene Carbon		
52	0.6	0.394 (9)	0.333 (20)	0.670 (13)
53	0.6	0.308 (13)	0.250 (30)	0.648 (19)
54	0.6	0.343 (13)	0.667 (30)	0.758 (19)
55	0.6	0.447 (13)	0.500 (30)	0.870 (19)
56	0.6	0.400 (9)	0.222 (20)	0.866 (13)
57	0.6	0.344 (9)	0.292 (20)	0.948 (13)
58	0.4	0.433 (13)	0.444 (30)	0.705 (19)
59	0.4	0.374 (13)	0.653 (30)	0.751 (19)
60	0.4	0.330 (13)	0.354 (30)	0.950 (19)
61	0.4	0.423 (13)	0.361 (30)	0.871 (19)
62	0.4	0.278 (13)	0.208 (30)	0.640 (19)
63	0.4	0.396 (13)	0.153 (30)	0.744 (19)
		Attached to Methyl Carbon		
64	1.0	0.241 (5)	0.532 (10)	0.545 (6)
65	0.4	0.343 (13)	0.593 (30)	0.525 (19)
66	0.6	0.334 (9)	0.657 (20)	0.578 (13)
67	0.6	0.309 (9)	0.433 (20)	0.479 (13)
68	0.4	0.293 (13)	0.396 (30)	0.542 (19)
69	1.0	0.308 (5)	0.683 (10)	0.911 (6)
70	0.4	0.418 (13)	0.625 (30)	0.948 (19)
71	0.6	0.396 (9)	0.778 (20)	0.969 (13)
72	0.4	0.400 (13)	0.740 (30)	0.876 (19)
73	0.6	0.389 (9)	0.597 (20)	1.035 (13)
74	1.0	0.226 (5)	0.153 (10)	0.815 (6)
75	0.6	0.291 (9)	0.021 (20)	0.901 (13)
76	0.6	0.295 (13)	0.021 (30)	0.752 (19)
77	0.4	0.337 (13)	0.083 (30)	0.890 (19)
78	0.4	0.262 (13)	0.021 (30)	0.765 (19)

[a] Values in parentheses are standard deviations from least-squares refinement for H(40) and H(50). For other hydrogen atoms the standard deviations are guesses, based upon the appearance and disorder, set equal to 1, 2, or 3 times these values for H(40) and H(50). [b] Isotropic temperature factors were set at 3.0 for H(51), 4.0 for H(31)–H(50) and H(52)-H(63), and 5.0 for H(64)–H(78).

(13) R. D. Dobrott and W. N. Lipscomb, *J. Chem. Phys.*, **37**, 1779 (1962).

TABLE V
RMS AMPLITUDES AND DIRECTION COSINES[a] OF THERMAL MOTION

Atom	B	Rms amp, Å	Cos A	Cos B	Cos C	Atom	B	Rms amp, Å	Cos A	Cos B	Cos C
B(1)	6.21	0.280	0.446	0.877	0.018	B(15)	5.24	0.258	−0.840	−0.308	0.703
	4.07	0.227	−0.894	0.446	0.341		4.22	0.231	−0.357	0.933	0.093
	2.53	0.178	−0.044	−0.179	0.940		2.66	0.183	0.407	0.184	0.705
B(2)	5.16	0.256	−0.969	0.223	0.228	B(16)	4.09	0.228	−0.338	0.890	0.402
	3.81	0.220	−0.232	−0.688	0.726		3.17	0.200	−0.832	−0.435	0.605
	3.22	0.202	0.081	0.691	0.649		2.82	0.189	0.440	−0.138	0.687
B(3)	5.14	0.255	0.795	0.182	0.276	B(18)	5.00	0.252	0.293	−0.639	0.570
	3.39	0.207	0.108	−0.981	0.114		3.69	0.216	0.401	0.757	0.350
	2.22	0.168	−0.596	0.065	0.954		2.62	0.182	−0.868	0.134	0.743
B(4)	5.81	0.271	0.620	−0.648	0.207	B(19)	4.88	0.249	−0.149	0.962	0.265
	4.02	0.226	−0.664	−0.734	0.088		3.36	0.206	−0.980	−0.174	0.419
	2.87	0.191	−0.418	0.204	0.974		2.59	0.181	0.130	−0.210	0.868
B(5)	4.81	0.247	0.075	−0.469	0.803	B(20)	4.89	0.249	−0.497	−0.862	0.075
	4.44	0.237	0.533	0.764	0.162		3.40	0.207	−0.831	0.441	0.599
	2.43	0.175	−0.843	0.442	0.573		2.91	0.192	0.248	−0.249	0.797
B(6)	4.11	0.228	−0.267	−0.905	0.402	N(21)	4.51	0.239	−0.953	−0.285	0.416
	3.54	0.212	−0.922	0.340	0.485		3.85	0.221	−0.274	0.955	0.199
	2.26	0.169	0.281	0.256	0.776		3.06	0.197	0.128	−0.080	0.887
B(7)	5.76	0.270	−0.473	0.671	0.696	C(22)	9.07	0.339	0.592	0.767	0.034
	5.12	0.255	−0.306	−0.732	0.676		4.07	0.227	0.513	−0.596	0.409
	3.52	0.211	0.826	0.113	0.241		2.86	0.190	−0.622	0.238	0.912
B(8)	6.64	0.290	−0.139	−0.874	0.486	C(23)	10.88	0.371	−0.950	0.286	0.442
	3.82	0.220	0.612	0.294	0.485		6.38	0.284	−0.104	−0.676	0.722
	2.69	0.185	−0.779	0.387	0.727		3.58	0.213	0.295	0.679	0.533
B(9)	3.48	0.210	−0.347	0.898	0.372	C(24)	7.44	0.307	−0.968	−0.250	0.297
	2.93	0.193	−0.937	−0.320	0.186		5.17	0.256	−0.235	0.853	0.519
	2.55	0.180	−0.037	−0.302	0.909		2.76	0.187	0.090	−0.459	0.802
B(10)	4.58	0.241	−0.443	−0.842	0.438	C(25)	9.82	0.353	−0.386	0.902	0.314
	4.03	0.226	0.878	−0.340	0.020		6.05	0.277	0.649	0.115	0.489
	2.36	0.173	−0.179	0.418	0.899		3.21	0.201	−0.656	−0.417	0.814
B(11)	9.32	0.343	−0.503	0.785	0.510	C(26)	10.46	0.364	−0.982	0.182	0.369
	4.72	0.244	−0.859	−0.501	0.189		10.06	0.357	−0.129	−0.819	0.571
	3.63	0.214	0.097	−0.364	0.839		2.75	0.187	0.135	0.545	0.734
B(12)	2.88	0.191	−0.792	−0.165	0.820	C(27)	8.41	0.326	0.530	0.077	0.617
	2.33	0.172	0.565	−0.564	0.376		8.04	0.319	−0.834	−0.132	0.785
	1.88	0.154	0.232	0.809	0.431		3.46	0.209	−0.153	0.988	0.057
B(13)	6.19	0.280	−0.956	−0.079	0.056	C(28)	7.80	0.314	0.214	0.883	0.320
	4.95	0.250	−0.166	−0.651	0.753		5.52	0.264	−0.922	0.324	0.111
	3.12	0.199	−0.243	0.755	0.656		3.56	0.212	−0.323	−0.339	0.941
B(14)	6.13	0.279	−0.816	−0.366	0.697	C(29)	10.26	0.360	−0.998	0.019	0.393
	4.05	0.226	−0.385	0.921	0.177		9.19	0.341	0.025	−0.759	0.604
	2.82	0.189	0.431	0.132	0.695		5.32	0.259	0.058	0.651	0.693
						C(30)	8.29	0.324	−0.928	0.268	0.557
							7.29	0.304	0.346	0.356	0.701
							4.27	0.233	−0.140	−0.895	0.445

[a] Direction cosines of the three principal axes are given relative to the crystallographic axes.

TABLE VI
BOND DISTANCES (Å)

Cage Coupling[a]

9–9′	1.72 (1)	19–19′	1.65 (1)
10–9′	1.92 (1)	20–19′	1.86 (1)

Cage Apex–Edge

1–2	1.87 (1)	13–11	1.68 (2)
1–3	1.65 (1)	13–12	1.70 (2)
1–4	1.66 (1)	13–7	1.56 (1)
1–5	1.69 (1)	13–18	1.73 (1)
6–10	1.78 (1)	14–20	1.78 (1)
7–10	1.58 (1)	15–20	1.68 (1)
8–10	1.75 (1)	16–20	1.76 (1)
9–10	1.68 (1)	19–20	1.67 (1)

Cage Basal Edge–Edge

2–3	1.91 (2)	11–12	1.79 (1)
3–4	1.83 (1)	7–12	1.86 (1)
4–5	1.95 (1)	7–18	1.98 (1)
2–5	1.89 (1)	11–18	1.82 (1)
6–7	1.66 (1)	14–15	1.79 (1)
7–8	1.71 (1)	15–16	1.79 (1)
8–9	1.86 (1)	16–19	1.83 (1)
6–9	1.85 (1)	14–19	1.93 (1)

Nitrogen–Methylene Carbon

21–22	1.50 (1)	21–25	1.56 (1)
21–23	1.54 (1)	21–26	1.50 (1)
21–24	1.52 (1)	21–27	1.56 (1)

Methylene–Methyl Carbon

22–28	1.50 (1)	25–28	1.51 (1)
23–29	1.46 (1)	26–29	1.57 (1)
24–30	1.51 (1)	27–30	1.50 (1)

Nitrogen–Hydrogen

21–51	1.14 (9)

Methylene Carbon–Hydrogen

22–52	1.03 (16)	25–58	1.36 (24)
22–53	1.02 (23)	25–59	1.23 (22)
23–54	1.09 (22)	26–60	1.24 (23)
23–55	1.33 (23)	26–61	1.27 (23)
24–56	1.01 (16)	27–62	1.34 (21)
24–57	0.98 (15)	27–63	1.34 (24)

Cage Belt Edge–Edge

2–6	1.77 (1)	11–14	1.78 (1)
2–7	1.70 (1)	11–15	1.91 (2)
3–7	1.86 (1)	12–15	1.80 (1)
3–8	1.82 (1)	12–16	1.75 (1)
4–8	1.78 (1)	7–16	1.90 (1)
4–9	1.80 (1)	7–19	2.00 (1)
5–9	1.82 (1)	18–19	1.80 (1)
5–6	1.78 (1)	18–14	1.77 (1)

Terminal B–H

1–31	1.08 (15)	11–41	1.29 (15)
2–32	1.19 (16)	12–42	1.29 (15)
3–33	1.22 (16)	13–43	1.17 (15)
4–34	1.24 (15)	14–44	1.23 (15)
5–35	1.33 (16)	15–45	1.12 (16)
6–36	1.25 (16)	16–46	1.16 (16)
7–37	1.12 (22)	7–47	1.00 (23)
8–38	1.22 (17)	18–48	1.25 (16)
10–40	1.14 (8)	20–50	1.24 (7)

Methyl Carbon–Hydrogen

28–64	1.08 (9)		
28–66	1.27 (15)	28–65	1.16 (23)
28–67	1.15 (15)	28–68	0.85 (23)
29–69	0.98 (9)		
29–71	1.16 (15)	29–70	0.92 (24)
29–73	1.08 (14)	29–72	1.36 (23)
30–74	1.07 (9)		
30–75	1.06 (15)	30–77	0.96 (22)
30–76	1.16 (22)	30–78	0.98 (22)

[a] Primed atoms are related to unprimed atoms by a center of symmetry.

TABLE VII

BOND ANGLES (DEG)[a]

Cage to Cage[b]

9′–10–9	56 (1)	19′–20–19	56 (1)
9′–9–10	69 (1)	19′–19–20	68 (1)
10′–9–9′	55 (1)	20′–19–19′	57 (1)
10′–9–10	124 (1)	20′–19–20	124 (1)
9′–10–6	100 (1)	19′–20–14	102 (1)
9′–10–7	147 (1)	19′–20–15	154 (1)
9′–10–8	101 (1)	19′–20–16	102 (1)
9′–9–6	106 (1)	19′–19–14	104 (1)
9′–9–8	105 (1)	19′–19–16	108 (1)
9′–9–4	145 (1)	19′–19–7	152 (1)
9′–9–5	148 (1)	19′–19–18	144 (1)
10′–9–6	132 (1)	20′–19–14	133 (1)
10′–9–8	133 (1)	20′–19–16	136 (1)
10′–9–4	112 (1)	20′–19–7	113 (1)
10′–9–5	113 (1)	20′–19–18	112 (1)

Boron Cage Angles of Triangular Faces

2–1–3	65 (1)	11–13–12	64 (1)
3–1–4	67 (1)	12–13–7	69 (1)
4–1–5	71 (1)	7–13–18	74 (1)
5–1–2	64 (1)	18–13–11	65 (1)
1–2–3	52 (1)	13–11–12	58 (1)
3–2–7	62 (1)	12–11–15	58 (1)
7–2–6	57 (1)	15–11–14	58 (1)
6–2–5	58 (1)	14–11–18	59 (1)
5–2–1	53 (1)	18–11–13	59 (1)
1–3–2	63 (1)	13–12–11	58 (1)
2–3–7	54 (1)	11–12–15	65 (1)
7–3–8	55 (1)	15–12–16	61 (1)
8–3–4	58 (1)	16–12–7	63 (1)
4–3–1	57 (1)	7–12–13	52 (1)
1–4–3	56 (1)	13–7–12	59 (1)
3–4–8	60 (1)	12–7–16	56 (1)
8–4–9	63 (1)	16–7–19	56 (1)
9–4–5	58 (1)	19–7–18	54 (1)
5–4–1	55 (1)	18–7–13	57 (1)
1–5–4	54 (1)	13–18–7	49 (1)
4–5–9	57 (1)	7–18–19	64 (1)
9–5–6	62 (1)	19–18–14	65 (1)
6–5–2	58 (1)	14–18–11	59 (1)
2–5–1	63 (1)	11–18–13	56 (1)
10–6–7	55 (1)	20–14–15	56 (1)
7–6–2	59 (1)	15–14–11	65 (1)
2–6–5	64 (1)	11–14–18	62 (1)
5–6–9	60 (1)	18–14–19	58 (1)
9–6–10	55 (1)	19–14–20	53 (1)
10–7–8	64 (1)	20–15–16	61 (1)
8–7–3	61 (1)	16–15–12	58 (1)
3–7–2	65 (1)	12–15–11	57 (1)
2–7–6	64 (1)	11–15–14	57 (1)
6–7–10	66 (1)	14–15–20	62 (1)
10–8–9	55 (1)	20–16–19	55 (1)
9–8–4	59 (1)	19–16–7	65 (1)
4–8–3	61 (1)	7–16–12	61 (1)
3–8–7	64 (1)	12–16–15	61 (1)
7–8–10	54 (1)	15–16–20	56 (1)
10–9–6	60 (1)	20–19–14	59 (1)
6–9–5	58 (1)	14–19–18	57 (1)
5–9–4	65 (1)	18–19–7	63 (1)
4–9–8	58 (1)	7–19–16	59 (1)
8–9–10	59 (1)	16–19–20	60 (1)
6–10–7	59 (1)	14–20–15	62 (1)
7–10–8	61 (1)	15–20–16	63 (1)
8–10–9	66 (1)	16–20–19	64 (1)
9–10–6	64 (1)	19–20–14	68 (1)

Apical Hydrogen[b]

40–10–9′	96 (3)	50–20–19′	92 (4)
40–10–9	152 (3)	50–20–19	146 (4)
40–10–6	134 (4)	50–20–16	140 (4)
40–10–7	117 (3)	50–20–14	118 (4)
40–10–8	121 (4)	50–20–15	114 (3)
31–1–2	119 (8)	43–13–11	120 (7)
31–1–3	141 (8)	43–13–12	143 (8)
31–1–4	137 (8)	43–13–7	130 (7)
31–1–5	113 (8)	43–13–18	116 (8)

Sum of Angles at Each Vertex

1	267 (2)	13	272 (2)
10	250 (2)	20	257 (2)
2	282 (2)	11	292 (2)
3	287 (2)	12	299 (2)
4	292 (2)	7	282 (2)
5	294 (2)	18	293 (2)
6	293 (2)	14	294 (2)
7	320 (2)	15	295 (2)
8	293 (2)	16	298 (2)
9	300 (2)	19	298 (2)

Boron Cage Pyramid Base Angles

5–2–3	87 (1)	18–11–12	92 (1)
2–3–4	94 (1)	11–12–7	93 (1)
3–4–5	88 (1)	12–7–18	85 (1)
4–5–2	91 (1)	7–18–11	88 (1)
9–6–7	83 (1)	19–14–15	86 (1)
6–7–8	105 (1)	14–15–16	95 (1)
7–8–9	81 (1)	15–16–19	89 (1)
8–9–6	92 (1)	16–19–14	89 (1)

Boron Cage Equatorial Terminal Hydrogen Angles

32–2–1	125 (7)	41–11–13	124 (7)
32–2–3	138 (7)	41–11–12	128 (7)
32–2–5	127 (8)	41–11–18	136 (7)
32–2–6	115 (7)	41–11–14	118 (7)
32–2–7	129 (7)	41–11–15	117 (7)
33–3–1	127 (7)	42–12–13	114 (7)
33–3–2	133 (7)	42–12–11	135 (7)
33–3–4	132 (7)	42–12–7	117 (7)
33–3–7	123 (7)	42–12–15	127 (7)
33–3–8	113 (7)	42–12–16	117 (7)
34–4–1	135 (7)	47–7–13	143 (12)
34–4–3	133 (7)	47–7–12	130 (13)
34–4–5	139 (7)	47–7–18	144 (13)
34–4–8	111 (7)	47–7–16	100 (13)
34–4–9	110 (7)	47–7–19	109 (12)
35–5–1	123 (6)	48–8–13	128 (7)
35–5–2	134 (7)	48–18–11	134 (7)
35–5–4	131 (7)	48–18–7	133 (7)
35–5–6	117 (7)	48–18–14	113 (7)
35–5–9	117 (6)	48–18–19	113 (7)
36–6–2	115 (7)	44–14–11	126 (7)
36–6–5	112 (7)	44–14–18	124 (8)
36–6–7	142 (7)	44–14–15	129 (8)
36–6–9	132 (7)	44–14–19	129 (7)
36–6–10	125 (7)	44–14–20	112 (7)
37–7–2	111 (12)	45–15–11	129 (8)
37–7–3	112 (11)	45–15–12	120 (8)
37–7–6	128 (12)	45–15–14	134 (8)
37–7–8	122 (12)	45–15–16	123 (8)
37–7–10	111 (11)	45–15–20	112 (7)
38–8–3	110 (7)	46–16–12	121 (7)
38–8–4	113 (7)	46–16–7	122 (7)
38–8–7	138 (7)	46–16–15	125 (8)
38–8–9	138 (7)	46–16–19	133 (7)
38–8–10	127 (7)	46–16–20	114 (7)

TABLE VII (*Continued*)

Triethylammonium Skeleton			
22–21–23	107 (1)	25–21–26	110 (1)
22–21–24	112 (1)	25–21–27	109 (1)
23–21–24	112 (1)	26–21–27	107 (1)
21–22–28	116 (1)	21–25–28	111 (1)
21–23–29	113 (1)	21–26–29	109 (1)
21–24–30	113 (1)	21–27–30	111 (1)
Methylene Disorder			
22–21–25	46 (1)		
23–21–26	53 (1)		
24–21–27	50 (1)		
Unique Triethylammonium Hydrogens			
51–21–22	106 (3)	64–28–22	112 (4)
51–21–23	105 (4)	64–28–25	117 (4)
51–21–24	114 (4)	69–29–23	101 (4)
51–21–25	106 (4)	69–29–26	113 (4)
51–21–26	113 (3)	74–30–24	110 (4)
51–21–27	110 (4)	74–30–27	113 (4)
Disordered Triethylammonium Hydrogens			
52–22–21	115 (8)	58–25–21	108 (9)
52–22–28	110 (8)	58–25–28	110 (9)
52–22–53	101 (16)	58–25–59	106 (15)
53–22–21	98 (12)	59–25–21	97 (11)
53–22–28	117 (12)	59–25–28	123 (10)
54–23–21	97 (12)	60–26–21	94 (10)
54–23–29	108 (12)	60–26–29	108 (11)
54–23–55	136 (16)	60–26–61	116 (15)
55–23–21	98 (10)	61–26–21	78 (10)
55–23–29	103 (9)	61–26–29	135 (10)
56–24–21	102 (8)	62–27–21	98 (10)
56–24–30	109 (9)	62–27–30	108 (10)
56–24–57	115 (13)	62–27–63	101 (14)
57–24–21	113 (9)	63–27–21	115 (10)
57–24–30	105 (9)	63–27–30	120 (10)
66–28–22	120 (7)	65–28–25	96 (11)
66–28–64	100 (8)	65–28–64	120 (12)
66–28–67	113 (10)	65–28–68	124 (19)
67–28–22	104 (7)	68–28–25	111 (15)
67–28–64	107 (9)	68–28–64	91 (17)
71–29–23	111 (7)	70–29–26	73 (14)
71–29–69	98 (9)	70–29–69	160 (14)
71–29–73	91 (11)	70–29–72	58 (17)
73–29–23	132 (8)	72–29–26	106 (10)
73–29–69	119 (10)	72–29–69	102 (11)
75–30–24	111 (8)	77–30–27	92 (14)
75–30–74	97 (10)	77–30–74	142 (14)
75–30–76	106 (14)	77–30–78	120 (19)
76–30–24	115 (12)	78–30–27	95 (13)
76–30–74	117 (12)	78–30–74	87 (15)

[a] Equivalent atoms in the disorder are listed side by side. [b] Primed atoms are related to unprimed atoms by a center of symmetry.

[1.82 (2) Å in $Cu_2B_{10}H_{10}$]. Bond distances between the two cages are 1.72 (1) Å for 9–9′ and 1.65 (1) Å for 19–19′ across the center of symmetry and are 1.92 (1) Å for 10–9′ and 1.86 (1) Å for 20–19′ connecting the interior apex borons with the opposite cage. The most interesting result[14] is that interior apical hydrogens 40 and 50 are 1.14 (8) and 1.24 (7) Å from borons 10 and 20 but 2.34 (8) and 2.27 (8) Å from the nearest borons 9′ and 19′ in the opposite cage. The bond angles (40–10–9 and 50–20–19 in Table VII) within each B_{10} unit show that these hydrogens may even be displaced away from borons 9′ and 19′ in order to avoid the opposite cage. Thus the three-center B–B–B bridging is sufficient to account for the bonding between B_{10} units. Terminal B–H distances average to 1.20 (8) Å.

The two conformations of the triethylammonium ion are close to an idealized model having C_3 symmetry, in which all single bonds are perfectly staggered, and all methyl groups are the maximum distance from one another. A rotation of less than 15° about any N–C bond is sufficient to bring a skeletal model of C_3 symmetry into coincidence with the observed one. Thus, the triethylammonium ion resembles a propeller which may be pitched one way or the other as shown in Figure 5. Near invariants with respect to this disorder are the methyl carbons and nitrogen, ammonium hydrogen 51, and methyl hydrogens 64, 69, and 74 extending in the same direction. In Figure 5 these hydrogens are pictured above and partially overlapping N(21) and the methyl carbons. Thermal motions of the ion show consistent anisotropy, probably associated with its relatively flat shape. Large displacements are noted along the ion's local C_3 axis, which is close to the crystallographic x axis. In addition, ethyl group 22–25–28 has a large component of motion along the crystallographic y axis. The conformation and disorder observed here suggest that wherever the triethylammonium group approximates the C_3 conformation, a twofold disorder is likely. The packing of the ions in the unit cell is shown in Figure 6, in which for clarity we show only the black atoms of Figures 2–5 corresponding to one of the disordered sets of atoms. The shortest distances of nonbonded interaction are listed in Table VIII. Error values in parentheses are derived from errors in coordi-

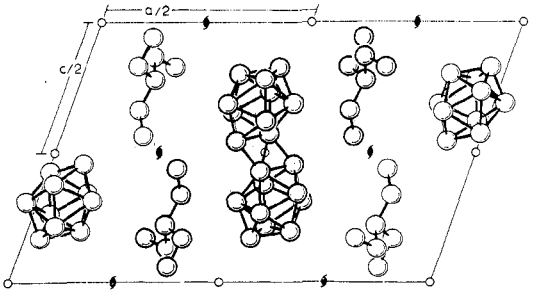

Figure 6.—The packing of ions in the unit cell.

(14) C. H. Schwalbe and W. N. Lipscomb, *J. Amer. Chem. Soc.*, **91**, 194 (1969).

TABLE VIII
SHORTEST INTER- AND INTRAIONIC NONBONDED CONTACTS (Å)[a]

Interionic[b]	
38i–63	1.8 (3)
50–52	2.0 (2)
46s–51	2.0 (2)
40i–52	2.0 (2)
48i–66	2.0 (3)
35s–64	2.1 (2)
33i–72	2.1 (3)
48i–59	2.2 (3)
32s–51	2.2 (2)
36s–51	2.2 (2)
34i–58	2.2 (3)
34i–65	2.2 (3)
8i–63	2.2 (2)
36s–62	2.2 (3)
34i–66	2.3 (2)
37i–58	2.3 (3)
40i–58	2.3 (2)
35–67	2.3 (2)
32s–74	2.3 (2)
42s–51	2.3 (2)
37i–63	2.3 (3)
47i–58	2.3 (3)
Intraionic	
Triethylammonium[c]	
C or N to H	
26–70	1.6 (2)
21–61	1.8 (2)
27–77	1.8 (2)
Triethylammonium	
C or N to H	
27–78	1.9 (2)
23–69	1.9 (1)
21–53	1.9 (2)
25–68	2.0 (2)
25–69	2.0 (2)
21–54	2.0 (2)
30–57	2.0 (2)
21–60	2.0 (2)
Intraionic	
Methylene[d] H to methyl or ammonium H	
59–72	1.5 (3)
62–68	1.9 (3)
54–66	2.1 (2)
54–69	2.1 (2)
58–61	2.1 (3)
61–63	2.1 (3)
62–78	2.1 (3)
Methyl and Methylene Group Internal Contacts[e]	
70–72	1.2 (3)
64–68	1.4 (2)
74–78	1.4 (2)
52–53	1.6 (3)
71–73	1.6 (2)
74–75	1.6 (2)
69–71	1.6 (2)

[a] Atoms transformed by symmetry operations are labeled with i for inversion or s for screw axis. [b] Estimated errors are in parentheses. The sum of van der Waals radii for H + H is about 2.4 Å. [c] The nonbonded C–H distance in staggered ethane is 2.2 Å. [d] The smallest H–H separation between methyl groups in staggered ethane is 2.5 Å. [e] In methane H–H is 1.8 Å.

nates and depend on guesses for unrefined hydrogens. Thus they are not standard deviations in the usual sense. Shortest contacts in each category involve hydrogens 63, 70, and 72, all of which were difficult to locate and occur at population 0.4. Other short interionic contacts may result from errors in H(52) and H(66) and from the fact that H(51), H(64), H(69), and H(74), though common to both disorder forms, need not be exactly superimposed. Since it is not possible to remove all short interionic contacts by an assumption of local ordering, the disorder is probably statistical in nature but with perhaps some short-range order. This conclusion is partly supported by a very slight amount of diffuseness along the *b* direction in the most intense reflections. For crystal structure studies requiring a positive ion approximating the size and shape of triethylammonium a better choice might be the N-ethylpyrrolidinium ion, in which two of the groups bonded to nitrogen are firmly tied together and therefore immune from this disorder.

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Crystal Structure of Tris(triethylammonium) μ-Nitrosobis(nonahydrodecaborate). The Structure of μ-Nitroso(nonahydrodecaborate)(3−)

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The $B_{20}H_{18}NO^{3-}$ anion, μ-nitrosobis(nonahydrodecaborate)(3−), consists of two $B_{10}H_9$ units each joined *via* an apical boron to the nitrogen atom of a bridging NO group. The approximate symmetry of this anion is C_{2v}. Some delocalization of electrons is suggested by the B–N bond distances of 1.51 (3) and 1.46 (3) Å and by the N–O distance of 1.28 (2) Å. A twofold disorder is present in one triethylammonium ion, which has an idealized nearly flat conformation of C_s symmetry. The two ordered triethylammonium ions each differ from C_3 symmetry by rotation of one ethyl group about a C–N bond. The monoclinic space group is $P2_1/n$, and there are four formula weights in a unit cell having parameters a = 24.44 (4), b = 16.68 (3), c = 9.65 (2) Å, β = 97.0 (2)°, ρ_{obsd} = 0.99 (2) g cm^{-3}, and ρ_{calcd} = 0.972 (4) g cm^{-3}. Nearly complete refinement has yielded $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.14$ for the 4555 X-ray maxima. The crystals are intensely purple, and the two visible absorption maxima are shifted toward the red in less polar solvents.

Polyhedral $B_{10}H_{10}^{2-}$ ion[1] undergoes oxidative coupling with NO_2 alone or with NO in the presence of Fe^{3+} to yield a product initially formulated[2] as $B_{14}H_{12}NO^{2-}$ but later shown[3] to be $B_{20}H_{18}NO^{3-}$. Its infrared ab-

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