bidentate ethylenediamine complexes.[7] The N–C distance of 1.477 (5) Å is equivalent to the value of 1.50 (2) Å reported for monomethylamine–boron trifluoride,[8] $(CH_3)H_2N \cdot BF_3$, and trimethylamine–boron trifluoride,[9] $(CH_3)_3N \cdot BF_3$. Although the variations are not statistically significant, some differences in bond lengths are expected due to differences in acceptor properties. The N–C distance also falls within the range 1.44–1.50 Å reported for a series of ethylenediamine–metal complexes.[6]

The N–B distance of 1.600 (7) Å may be compared with the values of 1.60 (2) Å ($H_3N \cdot BF_3$), 1.57 (2) Å ($(CH_3)H_2N \cdot BF_3$), and 1.58 (3) Å ($(CH_3)_3N \cdot BF_3$).[10] Methyl substituents increase molecular stability which is reflected in a shortening of the N–B distance; however, steric interaction in the trimethyl compound reverses this trend. Since BH_3 is a weaker acceptor than BF_3, the $N–BH_3$ distance should be longer than the corresponding $N–BF_3$. Although the bond lengths are not statistically different, the N–B distance in ethylenediamine–bisborane is longer than the distance in comparable BF_3 adducts.

The apparent average bond distances involving hydrogen are B–H = 1.10 Å, N–H = 0.85 Å, and C–H = 0.94 Å. The average angles are CNH = 110°, BNH = 108°, NCH = 107°, HCC = 110°, and NBH = 104°. The CNB angle is 114.9 (4)° and the CCN angle is 112.4 (4)°. All of these values are normal.

The ethylenediamine–bisborane molecule approximates C_{2h} symmetry in the solid state. This symmetry leads to 27 infrared-active fundamental vibrations, none of which is Raman active. Small deviations from C_{2h} symmetry may permit other vibrations to appear as weak bands. Since the infrared spectrum was assigned on the basis of a gauche conformation, a reanalysis of the spectrum is required.

Acknowledgment.—We wish to express our appreciation to the TCU Research Foundation for their financial assistance.

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[7] R. W. G. Wyckoff, "Crystal Structures," Vol. 5, Chapter XIV, Interscience, New York, N. Y., 1966.

[8] S. Geller and J. L. Hoard, *Acta Crystallogr.*, **3**, 121 (1950).

[9] S. Geller and J. L. Hoard, *ibid.*, **4**, 399 (1951).

[10] J. L. Hoard, S. Geller, and T. B. Owen, *ibid.*, **4**, 405 (1951).

CONTRIBUTION FROM THE LAWRENCE RADIATION LABORATORY AND DEPARTMENT OF CHEMISTRY,
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The Crystal Structure of Tetraethylammonium 2,2′-*commo*-Bis(nonahydrodicarba-2-cobalta-*closo*-decaborate), a Salt of the Cobalt Derivative of the Nonahydridodicarbanonaborate(2−) Ligand[1]

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The crystal structure of $(C_2H_5)_4NCo(B_7C_2H_9)_2$, the tetraethylammonium salt of 2,2′-*commo*-bis(nonahydrodicarba-2-cobalta-*closo*-decaborate)(1−), has been determined from X-ray diffraction data measured from a single crystal by counter methods. The crystals are tetragonal, space group $P4/n$, with $a = 16.556$ (6), $c = 8.580$ (4) Å, $Z = 4$, $d_x = 1.15$ g/cm³. Refinement by least squares reduced R to 0.097 for 926 reflections. The anion is a sandwich compound of cobalt with two carborane polyhedral fragments, in the shape of two distorted bicapped square antiprisms with one vertex (occupied by the cobalt atom) in common. Disorder exists for the location of half of the carbon atoms in the carborane cages and for the orientation of half of the tetraethylammonium ions. Average bond lengths (excluding atoms involved in disorder) are Co–C = 1.93, Co–B = 2.15, B–C (polar) = 1.60, B–B (polar) = 1.73, B–B (tropical) = 1.88, B–B (equatorial) = 1.83 Å.

Introduction

A new polyhedral transition metal complex containing the $B_7C_2H_9^{2-}$ ligand was prepared by Hawthorne and George,[2] who deduced its structure on the basis of nmr data and chemical analysis. The anion $Co(B_7C_2H_9)_2^-$, 2,2′-*commo*-bis(nonahydrodicarba-2-cobalta-*closo*-decaborate)(1−), is shown without hydrogen atoms in Figure 1. It is a sandwich compound of cobalt (formal oxidation state 3+) with two carborane polyhedral fragments. Carbon is in position 1 and in either position 6 or position 9 (Figure 1). Positions 6 and 9 are chemically equivalent in each cage but enantiomeric, and isomerism is possible depending on the relationship of the carbon atoms in the two cages of a complex.

This paper describes an X-ray diffraction analysis of the structure of tetragonal crystals of the tetraethylammonium salt of $Co(B_7C_2H_9)_2^-$. This study confirms the structural features found by Hawthorne and George[2] but fails to resolve the isomer problem because disorder is present. A monoclinic phase also present in the sample has not yet been fully characterized.

Experimental Section

Dark red crystals, provided by Professor Hawthorne, were a mixture of plates and needles. The plates, the subject of the present investigation, were flat prisms with {001} as the larger faces and {110} as the only other prominent form. Preliminary information was obtained from oscillation and Weissenberg photographs of a crystal mounted with [110] as the rotation axis, using Cu radiation. The systematic absences ($hk0$ absent if $h + k = 2n + 1$) were confirmed with a manually operated General Electric XRD-5 diffractometer with a quarter-circle Eulerian-cradle goniostat and a scintillation counter with pulse height discriminator. Mo radiation was used with a 0.003 in. thick Zr filter in front of the receiving slit. Cell dimensions were de-

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[1] Work done under the auspices of the U. S. Atomic Energy Commission

[2] M. F. Hawthorne and T. A. George, *J. Amer. Chem. Soc.*, **89**, 7114 (1967).

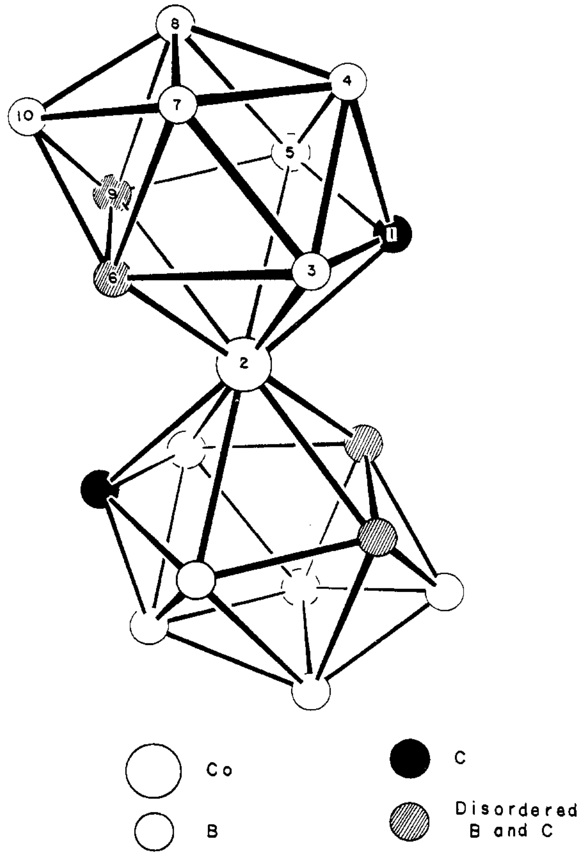

Figure 1.—Skeletal drawing of the $Co(B_7C_2H_9)_2^-$ anion.

rived from 2θ measurements of $hk0$ and $00l$ reflections for which $K\alpha_1$ and $K\alpha_2$ were resolved (λ 0.70926 Å for Mo $K\alpha_1$).

Intensity data were measured with the diffractometer using a second crystal, 0.30 × 0.26 × 0.09 mm in size, glued to a glass fiber with [100] as the rotation axis. The stationary-crystal, stationary-counter technique was employed. Each peak intensity was counted for 10 sec; then background was counted for 10 sec with 2θ offset about 1°. The standard deviation, $\sigma(I)$, of the net intensity was estimated according to the equation $\sigma^2(I) = I + 2B + (0.08I)^2$, where I is the peak count minus the background count and B is the background count. The factor 2 appears because the statistical variances of the peak count and background count are $I + B$ and B, respectively. The absorption coefficient of this crystal for Mo $K\alpha$ is estimated to be $\mu = 7.6\ \text{cm}^{-1}$. No correction was made for absorption. It is estimated that the extreme variation of the absorption factor for I is 15%.

All independent reflections with $2\theta \leq 45°$ ($(\sin\theta)/\lambda = 0.539$) were measured, but not all of them were used. Of the 473 reflections between $2\theta = 40$ and 45°, only the 146 with $I \geq 20$ counts were used. Excluding space group absences, the combination of these with all measurements at lower angles gave 1225 independent reflections of which 299 had $I < \sigma(I)$ and were assigned zero weight in the final refinement. Other reflections were assigned $w = [\sigma(F)]^{-2}$, where $\sigma(F) = k(I^{1/2} - [I - \sigma(I)]^{1/2})$; k is a scale factor divided by the square root of the Lorentz–polarization factor.

A CDC 6600 computer was used for all calculations. Refinement was accomplished with our unpublished full-matrix, least-squares program which minimizes the function $\Sigma w(\Delta F)^2/\Sigma w|F_o|^2$.

The atomic scattering factors of Cromer and Waber[3] for neutral atoms were used, with Cromer's corrections for anomalous dispersion[4] of the cobalt, $\Delta f' = 0.4$ and $\Delta f'' = 1.0$ electron. The stereoscopic illustration was prepared using Johnson's ORTEP program.[5]

The correlation of ΔF with intensity is poor, and no correction for extinction was made. It is estimated that, if the extinction effect is present, the intensity of the strongest reflection is reduced on the order of 12% and that only three reflections are reduced as much as 5%.

Crystal Data

The crystals are tetragonal with $a = 16.556$ (6) and $c = 8.580$ (4) Å at 24°. The density measured by flotation, 1.13 g/cm³, agrees with 1.15 g/cm³ calculated for four molecules of $[(C_2H_5)_4N][Co(B_7C_2H_9)_2]$ per cell. The Laue group is $4/m$ and reflections $hk0$ are absent if $h + k = 2n + 1$, which is characteristic of space group $P4/n$ (ref 6, no. 85). In this paper the structure is reported with the origin at $\bar{1}$ at $^1/_4$, $-^1/_4$, 0 from $\bar{4}$.

Space group $P4/nmm$, which has the same systematic absences, is ruled out by the Laue symmetry. Reasons for discarding lower symmetry space groups are discussed in the next section.

The monoclinic phase, the needles, have probable space group $P2_1/c$, with $a = 10.93$ (1), $b = 10.40$ (1), $c = 10.40$ (1) Å, and $\beta = 100.7$ (1)°. Their structure has not been determined.

Determination of the Structure

The structure determination was not straightforward.[7] The Co atoms were placed on the inversion centers 4(d), because this structure is consistent with the Patterson function and the expected structure of the anion.[8] A Fourier calculation including only the reflections with both h and k even (the only ones whose phases are controlled by Co) showed multiple images of the rest of the structure. Consideration of bond distances combined with some trial and error lead to the structure of the carborane cage. A Fourier map phased with all these atoms showed the N atoms at 2(b), site symmetry $\bar{4}$, and at 2(c) ($z \approx 0.5$), site symmetry 4. These locations were suggested earlier but had been disregarded

TABLE I

ATOMIC COORDINATES[a]

Atom	x	y	z
	Atoms in the $Co(B_7C_2H_9)_2^-$ Anion[b]		
Co(2)	0	0	0
C(1)	0.0891 (6)	0.0747 (6)	−0.0136 (13)
B(3)	0.0501 (8)	0.0907 (8)	0.1500 (17)
B(4)	0.0717 (8)	0.1716 (8)	0.0040 (18)
B(5)	0.0275 (8)	0.1016 (7)	−0.1472 (17)
BC(6)	−0.0571 (7)	0.0875 (7)	0.1281 (14)
B(7)	−0.0113 (9)	0.1872 (8)	0.1351 (18)
B(8)	−0.0243 (10)	0.1938 (9)	−0.0828 (18)
BC(9)	−0.0708 (7)	0.0945 (7)	−0.0715 (13)
B(10)	−0.1020 (9)	0.1682 (9)	0.0482 (19)
	Atoms in the Ordered Tetraethylammonium Group		
N(1)	$^1/_4$	$^3/_4$	$^1/_2$
C(11)	0.1985 (8)	−0.1983 (7)	0.3911 (13)
C(12)	0.1413 (10)	−0.1380 (11)	0.4792 (15)
	Atoms in the Disordered Tetraethylammonium Group		
N(2)	$^1/_4$	$^1/_4$	0.5299 (9)
C(13)[c]	0.2374 (13)	0.1807 (10)	0.4196 (19)
C(13)*[c]	0.2374	0.1807	0.6402
C(14)[c]	0.2247 (9)	0.0972 (7)	0.5299

[a] Standard deviations of the least significant digits estimated by least squares are given in parentheses. [b] The numbering system used for the anion is shown in Figure 1. [c] C(13) and C(13)* represent disordered half-atoms. Constraints imposed on the model of this disordered tetraethylammonium group are $x[C(13)] = x[C(13)^*]$, $y[C(13)] = y[C(13)^*]$, $z[C(13)] + z[C(13)^*] = 2z[N(2)]$, and $z[C(14)] = z[N(2)]$.

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(3) D. T. Cromer and J. T. Waber, *Acta Crystallogr.*, **18**, 104 (1965).

(4) D. T. Cromer, *ibid.*, **18**, 17 (1965).

(5) C. K. Johnson, "ORTEP, A Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustrations," Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1965.

(6) "International Tables for X-ray Crystallography," Vol. I, The Kynoch Press, Birmingham, England, 1952, p 175.

(7) A more complete description of the determination may be found in the Ph.D. Dissertation of D. St. Clair, University of California, Berkeley, Calif., 1969.

(8) Positions 4(e) are excluded without loss of generality. The alternative of 2(a) and 2(c) ($z = 0$) gives the same Patterson vectors, but it was never considered seriously. It leads to two nonequivalent anions, of symmetry $\bar{4}$ and 4, respectively, and forces all the tetramethylammonium ions to be centric. Such a model would involve even greater difficulties concerning disorder than does the structure reported here, and we believe that it is ruled out decisively by the subsequent calculations.

TABLE II

ANISOTROPIC THERMAL PARAMETERS,[a] Å^2

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Co(2)	3.6 (1)	3.7 (1)	5.3 (1)	−0.1 (1)	−0.1 (1)	−0.2 (1)
C(1)	4.5 (5)	3.4 (4)	7.3 (7)	−0.5 (4)	−0.3 (6)	−0.1 (6)
B(3)	6.0 (8)	4.0 (7)	7.7 (8)	0.2 (6)	−1.9 (7)	−0.4 (6)
B(4)	5.6 (7)	4.7 (7)	9.8 (10)	0.5 (5)	−1.4 (8)	0.0 (8)
B(5)	4.7 (7)	3.8 (6)	8.5 (9)	−0.3 (5)	1.5 (7)	0.3 (7)
BC(6)	6.6 (8)	5.0 (7)	6.5 (7)	−0.1 (6)	−0.1 (6)	−0.4 (6)
B(7)	7.4 (8)	3.8 (7)	9.1 (10)	−0.8 (6)	1.1 (8)	−0.6 (7)
B(8)	7.5 (10)	5.3 (8)	8.2 (9)	−1.4 (7)	−0.5 (8)	−0.3 (7)
BC(9)	4.0 (6)	3.8 (6)	5.8 (6)	−0.8 (4)	−0.3 (5)	0.1 (5)
B(10)	5.4 (8)	6.2 (9)	11.0 (11)	1.1 (7)	0.2 (8)	−0.2 (8)
N(1)[b]	6.8 (6)	6.8	3.9 (8)	0	0	0
C(11)	8.6 (8)	8.1 (8)	5.1 (6)	2.1 (7)	−0.7 (6)	0.8 (6)
C(12)	9.8 (10)	13.9 (13)	7.7 (9)	5.3 (9)	1.4 (7)	0.1 (8)
N(2)[b]	5.2 (5)	5.2	4.6 (8)	0	0	0
C(13)[c]	8.8 (12)	7.8 (9)	7.0 (9)	0.4 (10)	0.4 (11)	−4.0 (9)
C(13)*[c]	8.8	7.8	7.0	0.4	−0.4	4.0
C(14)	10.6 (9)	3.6 (5)	14.5 (10)	−0.5 (5)	−0.7 (7)	−2.0 (6)

[a] The temperature factors are $\exp(-\Sigma_3\Sigma_3 h_i h_j b_i b_j B_{ij}/4)$, where b_i is a reciprocal axis length. [b] Symmetry requires $B_{11} = B_{22}$, $B_{12} = B_{13} = B_{23} = 0$. [c] Constraints imposed on the model are B_{ij} of C(13)* equal, respectively, B_{11}, B_{22}, B_{33}, B_{12}, $-B_{13}$, $-B_{23}$ of C(13).

in our optimism that disorder could be avoided. The carbon atoms of the cation at 2(b) were clearly resolved, but at the fourfold axis we observed eight half-atoms for the α carbons and four larger peaks (at the same z coordinate as N) for the superposition of pairs of half-β-carbons. This result corresponds to the superposition of two tetraethylammonium ions, each of symmetry $\bar{4}$, and with relative rotation of 90° around the symmetry axis.

In the refinement by least squares, certain constraints are imposed on the coordinates and thermal parameters by the space group symmetry, as noted in Tables I and II. In order to avoid difficulties with an ill-conditioned model and a nearly singular matrix, we imposed some additional constraints on the disordered ethyl group, also as noted. These constraints prevent us from reaching the lowest possible residuals, though perhaps not by very much, and they spare us a great deal of ambiguity concerning alternate solutions.

In initial least-squares calculations, all atoms of the carborane cage were given boron form factors. Carbon atoms were then designated on the basis of the resulting thermal parameters and bond distances. A carbon atom assigned the scattering power of boron will have an abnormally low thermal parameter, and it is known that bond distances are in the order C–C < B–C < B–B for atoms having the same number of bonds. No assignment of carbon to just two positions is satisfactory, but one carbon at position 1 and the other disordered between positions 6 and 9 are consistent with the results. In further calculations, the atoms designated as BC(6) and BC(9) were given the average scattering power of boron and carbon.

In the final calculations, with anisotropic thermal parameters, $R = \Sigma|\Delta F|/\Sigma|F_o|$ was reduced to 0.097 for the 926 data included in the least squares and to 0.133 for 1225 data; $R_2 = [\Sigma w(\Delta F)^2/\Sigma w|F_o|^2]^{1/2}$ was 0.115. The standard deviation of an observation of unit weight was calculated as 1.9. Root-mean-square averages of $|\Delta F|/\sigma(F)$ taken over small intensity ranges indicated that the high-intensity reflections were overweighted by about a factor of 2. In the final cycle, no parameter shifted by more than 5% of its estimated standard deviation. A subsequent difference Fourier map showed a peak of magnitude 0.7 e/Å^3 at the cobalt position and other peaks 0.5 e/Å^3 or less, mostly near the carborane cage at distances too short to be hydrogen atoms. It was concluded that the data were inadequate to locate the hydrogen atoms.

Final values of the atomic coordinates are listed in Table I and of thermal parameters in Table II.[9]

When disorder is found, one seeks to avoid it by finding an alternative. In the present case there is no escape from disorder if one retains space group $P4/n$. The only space groups which permit ordered structures for these molecules with the same positions for Co and N are $P\bar{4}$, $P2/n$, Pn, $P2$, $P\bar{1}$, and $P1$. In each case problems of pseudosymmetry would make a unique interpretation difficult if not impossible, and with the possible exception of $P\bar{4}$ twinning would be expected. Except in $P2/n$ and Pn, there would be the problem of avoiding violations of the glide-plane extinction rules. The assumption of twinning of a structure in $P4/n$ offers no help in avoiding disorder. For these reasons these alternatives were discarded.

Discussion

As deduced by Hawthorne and George,[2] the structure of the anion may be described in terms of the bicapped square antiprism. Such a polyhedron, if fully symmetric, has an $\bar{8}$ axis passing through two vertices (positions 1 and 10). These two vertices may be described as "polar," while the other eight may be referred to as "tropical," to borrow geographical terms. In the present compound, the anion consists of two such polyhedra linked by a cobalt atom which is simultaneously in a tropical position in both cages. Because the bond lengths to cobalt are longer than the others, the cages deviate considerably from the ideal shape. The dimensions of the anion correspond to point symmetry $2/m$ (C_{2h}) within the accuracy of the determination.

In a preliminary communication to Professor Hawthorne, we said the carbon atoms were at positions 1 and 9 in Figure 1, and he later published that statement.[2] However, further analysis caused us to conclude, as stated above, that one carbon is at 1 (a polar position) and the other is distributed between positions 6 and 9, the two tropical positions in the opposite hemisphere which are also adjacent to cobalt. The alternate positions for the second carbon atom define enantiomorphic isomers of the carborane fragment. If the two configurations are arbitrarily designated as D and L, then the entire anion may be one of three isomers: DD, LL, or DL. The first two are enantiomers of the syn isomer, while the third is the anti isomer referred to by Hawthorne and George.[2] If order prevailed, the crystallographic symmetry would require all molecules to be DL. With disorder, however, it is impossible from the X-ray data to distinguish a mixture of DD and LL molecules from a mixture of orientations of DL molecules (*i.e.*, DL and LD), nor indeed can we exclude a mixture of DD, LL, DL, and LD. Thus we can make no definite conclusion concerning which isomers are present in the crystal.

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[9] A listing of structure factor amplitudes will appear immediately following this article in the microfilm edition of this volume of the journal. Single copies may be obtained from the Business Operations Office, Books and Journals Division, American Chemical Society, 1155 Sixteenth St., N.W., Washington, D. C. 20036, by referring to author, title of article, volume, and page number. Remit check or money order for $3.00 for photocopy or $2.00 for microfiche.

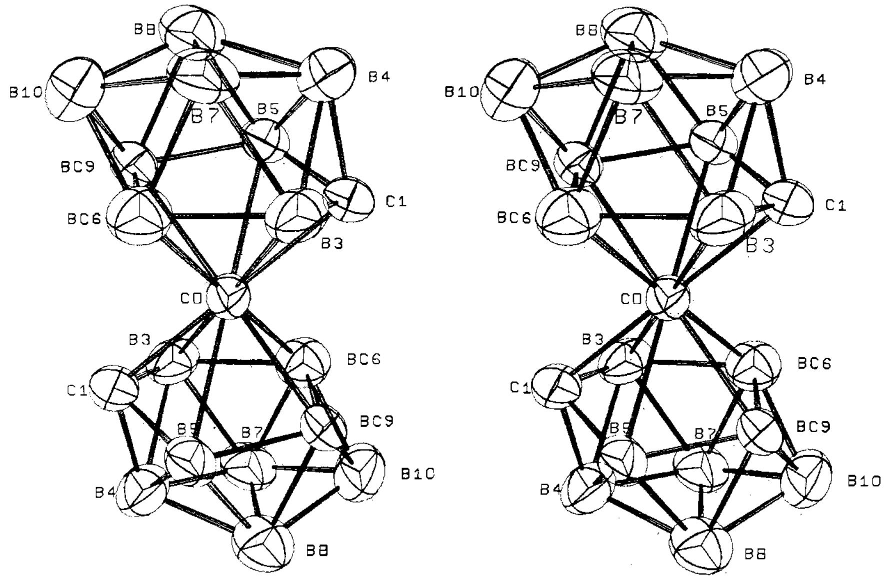

Figure 2. Steroscopic drawing of the $Co(B_7C_2H_9)_2^-$ anion showing thermal motion. Hydrogen atoms are not shown.

As described above, the carbon positions were deduced from bond distances and isotropic thermal parameters. Further evidence of disorder comes from the anisotropic thermal parameters. Because B–C bonds are shorter than B–B bonds, the atomic positions must be slightly different depending on which isomer is present at a given site. Thus the coordinates found by the least-squares analysis are not the actual positions of atoms but the averages of the various coordinates for the various molecules. The thermal amplitudes listed in Table III and depicted in Figure 2 do not correspond to any clear pattern of collective motion. We interpret them as representing a combination of the effects of thermal motion and of displacement of atoms according to the disorder.

TABLE III

PRINCIPAL VIBRATIONAL AMPLITUDES FOR THE $Co(B_7C_2H_9)_2^-$ ANION

Atom	Rms displacements, Å			Atom	Rms displacements, Å		
Co(2)	0.21	0.22	0.26	BC(6)	0.25	0.29	0.29
C(1)	0.20	0.24	0.30	B(7)	0.21	0.30	0.35
B(3)	0.22	0.25	0.34	B(8)	0.24	0.32	0.33
B(4)	0.24	0.26	0.36	BC(9)	0.20	0.24	0.27
B(5)	0.21	0.24	0.34	B(10)	0.24	0.30	0.37

TABLE IV

INTERATOMIC DISTANCES[a]

Atoms	Distance, Å	Atoms	Distance, Å
Atoms in the $Co(B_7C_2H_9)_2^-$ Anion			
Co(2)–C(1)	1.93	B(4)–B(7)	1.79
Co(2)–BC(9)	2.05	B(4)–B(5)	1.89
Co(2)–BC(6)	2.05	B(5)–BC(9)	1.76
Co(2)–B(3)	2.15	B(5)–B(8)	1.84
Co(2)–B(5)	2.15	BC(6)–BC(9)	1.73
C(1)–B(3)	1.57	BC(6)–B(7)	1.82
C(1)–B(5)	1.60	B(7)–B(8)	1.89
C(1)–B(4)	1.64	B(8)–BC(9)	1.82
B(3)–BC(6)	1.79	B(10)–BC(6)	1.68
B(3)–B(4)	1.87	B(10)–BC(9)	1.68
B(3)–B(7)	1.90	B(10)–B(7)	1.71
B(4)–B(8)	1.79	B(10)–B(8)	1.76
Atoms in the Ordered Tetraethylammonium Group			
N(1)–C(11)	1.53	C(11)–C(12)	1.57
Atoms in the Disordered Tetraethylammonium Group			
N(2)–C(13)	1.50	C(13)–C(14)	1.69

[a] Standard deviations estimated by least squares 0.01 Å for distances to Co(2) and 0.02 Å for all other distances.

TABLE V

BOND ANGLES IN THE $Co(B_7C_2H_9)_2^-$ ANION

Atoms	Angle, deg	Atoms	Angle, deg
Angles around Three-Membered Rings			
C(1)–Co(2)–B(3)	44.9 (5)	C(1)–Co(2)–B(5)	45.7 (5)
B(3)–Co(2)–BC(6)	50.3 (5)	B(5)–Co(2)–BC(9)	49.3 (5)
BC(6)–Co(2)–BC(9)	50.0 (5)	B(3)–C(1)–B(4)	71.3 (8)
B(4)–C(1)–B(5)	71.4 (8)	C(1)–B(3)–B(4)	56.1 (7)
C(1)–B(4)–B(3)	52.6 (7)	C(1)–B(4)–B(5)	53.4 (7)
C(1)–B(5)–B(4)	55.3 (7)	B(3)–B(4)–B(7)	62.3 (8)
B(4)–B(3)–B(7)	56.9 (7)	B(3)–B(7)–B(4)	60.7 (8)
B(3)–B(7)–BC(6)	57.4 (7)	B(3)–BC(6)–B(7)	63.6 (7)
BC(6)–B(3)–B(7)	59.0 (7)	BC(6)–B(7)–B(10)	56.7 (8)
B(7)–BC(6)–B(10)	58.3 (8)	BC(6)–B(10)–B(7)	65.0 (8)
BC(6)–BC(9)–B(10)	58.9 (8)	BC(9)–BC(6)–B(10)	58.9 (8)
BC(6)–B(10)–BC(9)	62.2 (8)	B(8)–B(10)–BC(9)	63.8 (8)
B(8)–BC(9)–B(10)	60.3 (8)	BC(9)–B(8)–B(10)	55.9 (8)
B(5)–B(8)–BC(9)	57.4 (7)	B(5)–BC(9)–B(8)	61.8 (7)
B(8)–B(5)–BC(9)	60.7 (7)	B(5)–B(4)–B(8)	59.8 (8)
B(4)–B(5)–B(8)	57.6 (8)	B(4)–B(8)–B(5)	62.6 (8)
B(4)–B(7)–B(8)	58.3 (8)	B(7)–B(4)–B(8)	63.4 (8)
B(4)–B(8)–B(7)	58.4 (8)	B(7)–B(8)–B(10)	55.7 (8)
B(8)–B(7)–B(10)	58.4 (9)	B(7)–B(10)–B(8)	65.9 (9)
Angles around Four-Membered Rings			
Co(2)–B(3)–B(4)	100.0 (8)	Co(2)–B(5)–B(4)	99.1 (8)
B(3)–Co(2)–B(5)	73.9 (5)	B(3)–B(4)–B(5)	87.0 (8)
BC(6)–BC(9)–B(8)	88.4 (9)	B(7)–B(8)–BC(9)	86.7 (8)
BC(6)–BC(9)–B(8)	93.3 (8)	B(7)–BC(6)–BC(9)	91.5 (8)
Angles around Five-Membered Rings			
C(1)–B(3)–BC(6)	108.0 (10)	C(1)–B(5)–BC(9)	107.9 (9)
B(3)–BC(6)–BC(9)	103.5 (9)	B(5)–BC(9)–BC(6)	104.4 (8)
B(3)–C(1)–B(5)	109.5 (9)	C(1)–Co(2)–BC(6)	86.1 (5)
C(1)–B(4)–B(7)	109.5 (9)	Co(2)–BC(6)–B(7)	117.9 (8)
B(4)–B(7)–BC(6)	99.7 (9)	Co(2)–C(1)–B(4)	119.3 (7)
C(1)–B(3)–B(7)	107.8 (10)	C(1)–B(5)–B(8)	108.3 (10)
B(3)–B(7)–B(8)	100.2 (9)	B(5)–B(8)–B(7)	101.4 (9)
C(1)–Co(2)–BC(9)	85.9 (4)	C(1)–B(4)–B(8)	108.6 (10)
B(4)–B(8)–BC(9)	99.7 (9)	Co(2)–BC(9)–B(8)	117.7 (7)
Co(2)–B(3)–B(7)	110.0 (8)	Co(2)–BC(9)–B(10)	123.3 (8)
B(3)–Co(2)–BC(9)	82.3 (5)	B(3)–B(7)–B(10)	110.3 (10)
B(7)–B(10)–BC(9)	97.5 (10)	B(3)–B(4)–B(8)	104.8 (9)
B(3)–BC(6)–B(10)	117.4 (9)	B(4)–B(3)–BC(6)	98.1 (9)
BC(6)–B(10)–B(8)	97.4 (10)	B(4)–B(8)–B(10)	109.5 (11)
B(4)–B(5)–BC(9)	98.4 (9)	B(4)–B(7)–B(10)	111.9 (11)
B(5)–B(4)–B(7)	102.8 (9)	B(5)–BC(9)–B(10)	117.6 (9)
Co(2)–B(5)–B(8)	112.0 (8)	Co(2)–BC(6)–B(10)	123.3 (9)
B(5)–Co(2)–BC(6)	81.9 (5)	B(5)–B(8)–B(10)	109.5 (10)

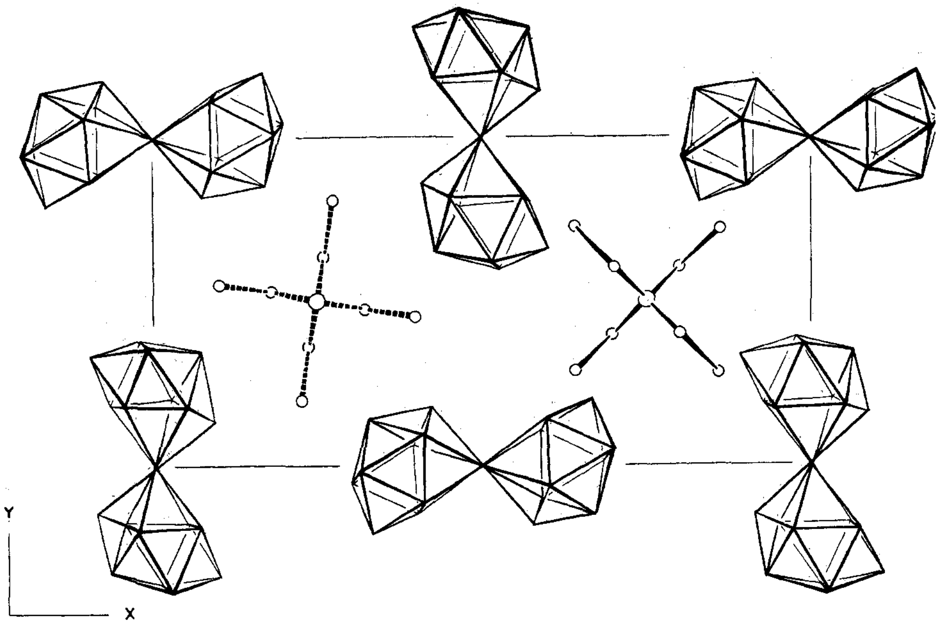

Figure 3.—View of half of the unit cell (x, 0–1; y, 0–$^1/_2$) looking down the c axis of the unit cell. The cobalt atoms are at $z = 0$ and the nitrogen atoms are near $z = {}^1/_2$. The ordered tetraethylammonium group is shown with solid bonds and the disordered group is shown with dashed bonds.

Bond distances and angles are listed in Tables IV and V. Because of the disorder, caution should be exercised in the use of the values involving BC(6), BC(9), C(13), and C(14). The standard deviations quoted are derived from the least-squares results without including any allowance for the disorder.

We designate bonds as "polar" if they involve an atom in a polar site, "tropical" if they are between atoms in the same square, and "equatorial" if they involve atoms in different squares. The bond lengths between atoms which are unambiguously identified as boron average 1.73 (3) Å for polar, 1.88 (1) Å for tropical, and 1.83 (5) Å for equatorial bonds, where the standard deviations indicate the scatter. These values are in excellent agreement with averages of similar types of bonds in $Cu_2B_{10}H_{10}$ and in $B_{20}H_{18}NO_3^{3-}$: polar B–B = 1.72 Å, tropical B–B = 1.87 Å, equatorial B–B = 1.82 Å.[10,11] These lengths may be compared with B–B = 1.78 Å in typical icosahedral carborane structures.[12] The shorter bond lengths to the polar atom reflect its lower number of neighbors and corresponding higher bond order. A molecular orbital explanation has been given for the greater length of the tropical bonds.[10] The three polar B–C bonds in our compound average 1.60 (4) Å, which is also significantly shorter than B–C = 1.71 Å in an icosahedral carborane.[12]

The molecular packing shown in Figure 3 may be described as a CsCl-type packing in a pseudocubic tetragonal pseudocell with $a' = a/2 = 8.28$ Å and $c' = c = 8.58$ Å. Such a structure is very favorable for the Coulomb interactions of a salt.

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