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David St. Clair, Allan Zalkin, and David H. Templeton

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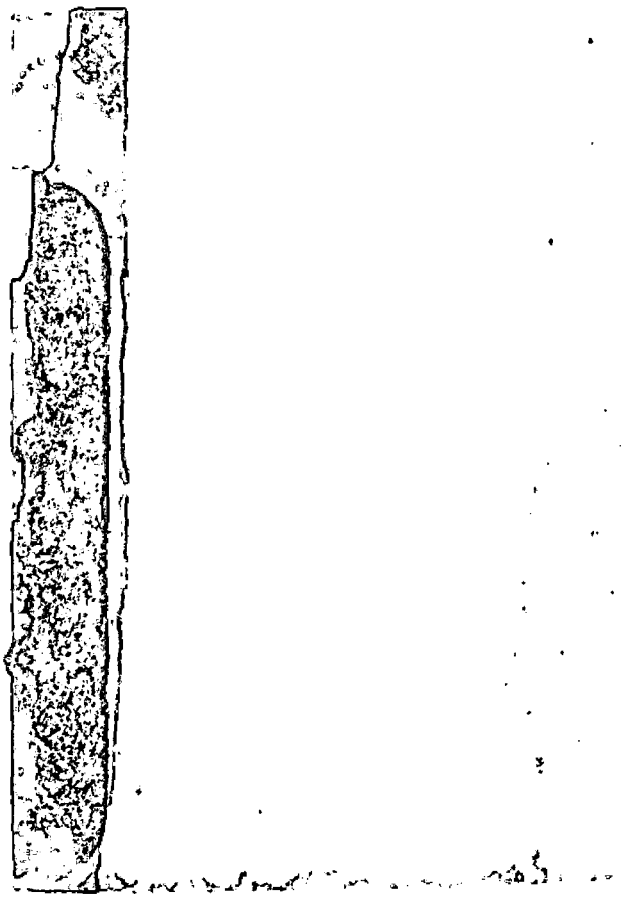

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UNIVERSITY OF CALIFORNIA

Lawrence Radiation Laboratory
Berkeley, California 94720

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CONTRIBUTION FROM THE LAWRENCE RADIATION LABORATORY AND DEPARTMENT OF CHEMISTRY, UNIVERSITY OF CALIFORNIA, BERKELEY, CALIFORNIA 94720

The Crystal Structure of $(C_2H_5)_4NCo(B_7C_2H_9)_2$, a Salt of the Cobalt Derivative of the $(B_7C_2H_9)^{-2}$ Carborane Ligand[1]

By David St. Clair, Allan Zalkin, and David H. Templeton

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(1) Work done under the auspices of the U. S. Atomic Energy Commission.

Abstract

The crystal structure of the tetraethylammonium salt of the $Co(B_7C_2H_9)_2^-$ complex, 2,2'-commo-bis[nonahydro-dicarba-2-cobalta-closo-decaborate](1-), has been determined from 926 independent X-ray intensity data measured from a single-crystal by counter methods. The crystals are tetragonal, space group P4/n, with a = 16.556 Å and c = 8.580 Å. There are four molecules in the unit cell and the calculated density is ρ = 1.15 g/cc. The structure was refined by least squares to a conventional R value of 9.6 %. The anion, $Co(B_7C_2H_9)_2^-$, has the shape of two distorted bicapped square antiprisms which have one corner in common. This shared corner is occupied by the cobalt atom which lies on a crystallographic inversion center. The dimensions of the anion correspond to point symmetry C_{2h} (2/m) within the accuracy of the determination. Half of the $(C_2H_5)_4N^+$ cations lie on special positions having 4-fold rotational point symmetry and are disordered. One of the carbon atoms in each B_7C_2 fragment is located at an apex position but the other is disordered between two positions. Very anisotropic thermal parameters were found for most of the atoms. Their meaning is discussed in terms of disorder instead of thermal motion.

Introduction

A new polyhedral transition metal complex containing the $(B_7C_2H_9)^{-2}$ ligand has been prepared.[2] The anion, $Co(B_7C_2H_9)_2^-$, 2,2'-commo-bis

(2) M. F. Hawthorne and T. A. George, J. Am. Chem. Soc., 89, 7114(1967).

[nonahydro-dicarba-2-cobalta-closo-decaborate](1-), is shown without hydrogens in Figure 1. It is composed of a cobalt ion, of formal charge +3, sandwiched between the two $(B_7C_2H_9)^{-2}$ polyhedral fragments. The structure of the anion, proposed by Hawthorne and George[2] from NMR data, was based on the bicapped square antiprism geometry of $B_{10}H_{10}$ distorted to accommodate one cobalt and two carbon atoms. They concluded that both of the carbon atoms in both of the cages were bonded to the cobalt which was attached to an open 5-fold face. In each cage, there was one carbon occupying an apex position, labeled position number 1 in Figure 1, and the other carbon was located at an opposite corner of the 5-fold face. Since there are two equivalent positions for this second carbon in each cage, labeled positions number 6 and 9 in Figure 1, which fulfill this structural requirement, the possibility of isomers exists. The form in which the cobalt lies on an inversion center was called the antiisomer and the form in which the cobalt lies on a 2-fold rotation axis perpendicular to the plane containing positions 1, 2, 4 and 10 was called the synisomer.

This paper describes an X-ray structure analysis carried out using a single crystal of the tetraethylammonium salt of $Co(B_7C_2H_9)_2^-$. Because disorder was found, the accuracy of the determination was limited. The

gross structure proposed by Hawthorne and George was confirmed. One carbon is located at an apex, position 1, but the other carbon is disordered between positions 6 and 9 in each cage. Thus, no conclusion can be drawn about the isomerism of any single molecule.

Experimental

The dark red crystals of $(C_2H_5)_4NCo(B_7C_2H_9)_2$ sent to us by Professor Hawthorne, University of California, Riverside, grow in the shape of plate-like prisms with the (001) as the largest faces. The only other prominent faces are the (110) faces.

Two crystals were used in this analysis. The first crystal was glued to the tip of a glass fiber in air with its [110] direction parallel to the rotation axis of the fiber. From oscillation and Weissenberg photographs made using Cu radiation (Cu $K\alpha_1$; λ = 1.5405 Å), the space group and approximate cell dimensions were determined. Using a manually operated General Electric XRD-5 diffractometer with a molybdenum X-ray source (Mo $K\alpha_1$; λ = 0.70926 Å), the $\underline{hk}0$, $\underline{h} + \underline{k} = 2\underline{n}$ extinction rule was confirmed and from measurements on the $\underline{hk}0$, $\underline{h}\bar{\underline{k}}0$ and $00\underline{\ell}$ reflections, accurate cell dimensions were determined.

A second crystal, 0.30 x 0.26 x 0.09 mm, was glued to a glass fiber in air with its $\underline{a}$ axis parallel to the rotation axis of the fiber. Intensity data were measured using a manually operated General Electric XRD-5 diffractometer with a quarter circle, Eulerian cradle goniostat and a scintillation counter equipped with a pulse height discriminator. Molybdenum $K\alpha$ radiation was used with a 0.003 inch thick Zr filter placed in front of the receiving slit. The intensity of a reflection is the peak intensity minus the background intensity each counted for ten seconds with the crystal and counter stationary. The standard deviation, $\sigma(\underline{I})$, of the intensity was estimated according to the equation, $\sigma^2(\underline{I}) = \underline{I} + 2\underline{I}_b + (0.08\underline{I})^2$, where $\underline{I}$ is the count for the net intensity and $\underline{I}_b$ is the background count. The absorption coefficient

of this crystal for molybdenum X-rays is $\mu = 7.6\ cm^{-1}$. No correction for absorption or extinction was applied to the data. It is estimated that absorption effects could cause a maximum error of 7 % in the intensity data.

All independent reflections in one symmetry non-equivalent set with $2\theta \leqq 45^{\circ}$ ($\sin\theta/\lambda = 0.539$) were measured but not all of them were used in this work. Of the 473 data between $2\theta = 40^{\circ}$ and $2\theta = 45^{\circ}$, only the 146 with $\underline{I} \geqq 2$ counts/second were used. Excluding space group absences, the combination of these data with all the data below $2\theta \leqq 40^{\circ}$ gave 1225 independent reflections of which 299 had $\underline{I} < \sigma(\underline{I})$.

All calculations were done on a CDC 6600 computer. Lorentz and polarization corrections were applied and the data were converted into structure factors. Refinements were done with our unpublished version of a full matrix least squares program which minimizes the function $\Sigma \underline{w}(|\underline{F}_o| - |\underline{F}_c|)^2/\Sigma \underline{w}|\underline{F}_o|^2$, where $\underline{F}_o$ and $\underline{F}_c$ are the observed and calculated structure factors, respectively. The weighting factors, $\underline{w}$, were set equal to unity in the early refinements, but in the final refinements, $\underline{w} = [\sigma(\underline{F})]^{-2}$ was used. If $\underline{I} \leqq \sigma(\underline{I})$, then $\sigma(\underline{F}) = [\underline{LP}\sigma(\underline{I})]^{\frac{1}{2}}$ and if $\underline{I} > \sigma(\underline{I})$, then $\sigma(\underline{F}) = [\underline{LPI}]^{\frac{1}{2}} - [\underline{LPI} - \underline{LP}\sigma(\underline{I})]^{\frac{1}{2}}$, where $\underline{LP}$ is the Lorentz-polarization correction. Anisotropic temperature factors were of the form $\exp(-\underline{h}^2\beta_{11}-\underline{k}^2\beta_{22}-\underline{\ell}^2\beta_{33}-2\underline{hk}\beta_{12}-2\underline{h\ell}\beta_{13}-2\underline{k\ell}\beta_{23})$ for all atoms except the nitrogens for which the form was $\exp(-(\underline{h}^2 + \underline{k}^2)\beta_{11} - \underline{\ell}^2\beta_{33})$. The atomic scattering factors of Cromer and Waber[3] were used for all atoms

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

considering them to be in their neutral valence state. Cromer's

corrections for anomalous dispersion[4] applied to the scattering factors

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(4) D. T. Cromer, Acta Cryst., 18, 17(1965).

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of the cobalt are $\Delta f' = +0.4$ and $\Delta f'' = +1.0$ electrons. The stereoscopic pair drawing was prepared using Johnson's ORTEP program.[5]

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(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.

Crystal Data

The crystals are tetragonal with unit cell dimensions $\underline{a} = \underline{b} = 16.556 \pm 0.006$ Å and $\underline{c} = 8.580 \pm 0.004$ Å. The Laue group is 4/m and the only extinction rule is $\underline{hk}0$, $\underline{h} + \underline{k} = 2\underline{n}$, which is characteristic of the centric space group P4/n. With the origin of the unit cell at a center of symmetry, the general symmetry equivalent positions are $\pm(\underline{x},\underline{y},\underline{z};\ \frac{1}{2}-\underline{x},\frac{1}{2}-\underline{y},\underline{z};\ \frac{1}{2}-\underline{y},\underline{x},\underline{z};\ \underline{y},\frac{1}{2}-\underline{x},\underline{z})$. The density of the crystals, measured by flotation, is 1.13 g/cc and agrees with the density, ρ = 1.15 g/cc, calculated with four molecules in the unit cell.

Determination of the Structure

In space group P4/n, there are two choices of origin in common use. In this work, the origin was arbitrarily placed at a $\bar{1}$ position. The general symmetry equivalent positions in space group P4/n are eight fold, but there are only four molecules of $(C_2H_5)_4NCo(B_7C_2H_9)_2$ in the unit cell. Therefore, the cobalt and nitrogen atoms must lie on special positions. The remainder of the asymmetric unit, consisting of one complete boron-carbon cage and, in the absence of disorder, two distinct ethyl groups of the tetraethylammonium ions, lie on general positions.

Most of the intense reflections conform to the special relationship that $\underline{h} = 2\underline{n}$ and $\underline{k} = 2\underline{n}$, thus indicating that a substantial portion of the structure is near a $\bar{1}$ position. Therefore, the Co atoms were placed at special positions 4(d). This assignment was confirmed by the Co-Co Harker peaks which appeared on the Patterson function.

The four nitrogen atoms must lie on special positions which, in the absence of disorder, must have either $\bar{4}$ or 2-fold point symmetry to preserve the tetrahedral bonding geometry of a nitrogen in a tetraethylammonium group. Thus, in space group P4/n, there should be only two possible sets of special positions allowed for the nitrogens, either positions 2(a) and 2(b) or positions 4(f). According to the Co-N Patterson vectors, the z coordinate of the nitrogen atoms must be $\frac{1}{2}$ in which case the four positions 4(f) degenerate into the two positions of 2(b). These vectors rule out positions 2(a) at $\underline{z} = 0$. The Patterson would, however, allow two of the nitrogen atoms to be placed at positions 2(c) but the 4-fold point symmetry here would destroy the tetrahedral bonding geometry around these nitrogens unless the tetraethylammonium

groups were disordered. This disordered arrangement for two of the four tetraethylammonium groups was later confirmed.

The determination of the structure was not straight forward. Only the positions of the cobalts were known with certainty and since they were lying on inversion centers, they contributed phase information only for the reflections with h even and k even. A Fourier prepared using only the data having h even and k even conforms to a higher symmetry and contains many spurrious peaks as well as the peaks corresponding to atoms in the molecule. The structure was determined by comparing this Fourier with a model of the expected structure and by ruling out combinations of peaks as being chemically impossible. A complete description of the determination is presented elsewhere.[6]

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(6) D. St. Clair, Ph. D. Thesis, University of California, Berkeley, California, to be published.

A Fourier using all data and using phases calculated from all atoms except the tetraethylammonium groups was prepared. This clearly showed two tetrahedral tetraethylammonium groups whose nitrogens were lying on special positions 2(b). It also showed two other nitrogens at special positions 2(c) where z was slightly greater than $\frac{1}{2}$. These positions 2(c) must exhibit 4-fold rotational point symmetry along the z direction and as expected, the ethyl groups here appeared disordered. One carbon atom in the ethyl group, the one attached to the nitrogen, appeared on this Fourier as two resolved half atoms while the second carbon atom in the ethyl group, the one not attached to the nitrogen,

appeared as a single unresolved atom with about the same $\underline{z}$ coordinate as the nitrogen. This disorder can be described as two tetrahedral (more strictly speaking, sphenoidal) tetraethylammonium groups both with their $\bar{4}$ axis pointing in the $\underline{z}$ direction but one rotated around this axis by 90° from the position in which the two groups are coincident. Constraints required to preserve the sphenoidal symmetry of the disordered tetraethylammonium group were placed on the model refined by least squares.

When all the light atoms in the cage were given the scattering power of boron, a full matrix least squares refinement of the positions and anisotropic thermal parameters for the cobalt atom and positions and isotropic thermal parameters for all other atoms except hydrogens gave $\underline{R} = 0.124$ for 926 data, where $\underline{R} = \Sigma||\underline{F}_o| - |\underline{F}_c||/\Sigma|\underline{F}_o|$. Thermal parameters and interatomic distances showed that there was one carbon localized at position 1, an apex of the cage and that the other carbon was disordered between positions 6 and 9. Therefore, in all further refinements C(1) was given the scattering power of carbon and BC(6) and BC(9) were given the scattering power of half carbon and half boron.

A final series of refinement was done in which all atoms were given anisotropic thermal parameters, the anomalous dispersion corrections were included, and weights derived from the standard deviations were included for all reflections except those having $\underline{I} < \sigma(\underline{I})$ which were given zero weight. The resulting $\underline{R}$ value for 926 data with $\underline{I} \geq \sigma(\underline{I})$ was $\underline{R} = 0.096$, $\underline{R}$ calculated for all 1225 data was $\underline{R} = 0.133$ and the weighted $\underline{R}$ value was $\underline{R}_2 = 0.115$ where $\underline{R}_2 = [\Sigma\underline{w}(|\underline{F}_o| - |\underline{F}_c|)^2/\Sigma\underline{w}|\underline{F}_o|^2]^{\frac{1}{2}}$. The standard deviation of observation of unit weight, given by $[\Sigma\underline{w}(|\underline{F}_o| - |\underline{F}_c|)^2/(\underline{n} - \underline{p})]^{\frac{1}{2}}$ where $\underline{n}$ and $\underline{p}$ are the number of data and

parameters, respectively, was 1.9. Root mean square averages of $(|\underline{F}_o| - |\underline{F}_c|)/\sigma(\underline{F})$ taken over small intensity ranges indicated that the high intensity reflections were over-weighted by about a factor of two. In the final cycle of refinement, no parameter shifted by more than 10 % of its estimated standard deviation. A difference Fourier phased by the parameters resulting from the final refinement was prepared. The largest peak appeared at the origin and had a magnitude of 0.7 electron/$Å^3$. The next largest peak was 0.5 electron/$Å^3$ and the rest of the peaks decreased smoothly in peak height. Most of the residual electron density resided near the boron-carbon cage at distances too short to be hydrogen atoms. It was concluded that the data were inadequate to locate the hydrogen atoms.

Results and Discussion

Final values for the atomic coordinates of all atoms except hydrogens are given in Table I, final values for all anisotropic thermal parameters are given in Table II, and observed and calculated structure factors are given in Table III.

As expected by Hawthorne and George,[2] the structure of the $Co(B_7C_2H_9)_2^-$ anion is composed of two bicapped square antiprisms linked by a cobalt atom which simultaneously occupies a non-apical corner of each cage. The cages are considerably distorted from ideal square-antiprismatic shape. The dimensions of the anion correspond to point symmetry C_{2h} (2/m) within the accuracy of the determination.

In a preliminary communication to Professor Hawthorne, we said the carbon atoms were located at positions 1 and 9 in Figure 1 and he subsequently published that statement.[2] However, upon further investigation, we concluded that one carbon is localized at position 1, an apex, but the other carbon is disordered between positions 6 and 9. The effect of this disorder is most pronounced in the anisotropic thermal parameters. They are quite large and very anisotropic. Inspection of the r.m.s. thermal vibrational amplitudes of the atoms in the cage, given in Table IV, reveals very large displacements of the atoms. This result could, however, be accounted for in three ways; (1) the data are of poor quality and the temperature factors are meaningless because they are merely accommodating the large discrepancies between $\underline{F}_o$ and the correct values, (2) there actually is a large amount of very anisotropic thermal motion, or (3) for half of the cages in the lattice, a carbon occupies position 6 and a boron is in position 9, and for the rest of the cages, the positions of the carbon and boron are interchanged. For any given

Table I

Positional Parameters ($x10^4$)[a]

Atom	x	y	z
Atoms in the $Co(B_7C_2H_9)_2^-$ anion[b]			
Co(2)	0000(0)	0000(0)	0000(0)
C(1)	0891(6)	0747(6)	-0136(13)
B(3)	0501(8)	0907(8)	1500(17)
B(4)	0717(8)	1716(8)	0040(18)
B(5)	0275(8)	1016(7)	-1472(17)
BC(6)	-0571(7)	0875(7)	1281(14)
B(7)	-0113(9)	1872(8)	1351(18)
B(8)	-0243(10)	1938(9)	-0828(18)
BC(9)	-0708(7)	0945(7)	-0715(13)
B(10)	-1020(9)	1682(9)	0482(19)
Atoms in the ordered tetraethylammonium group			
N(1)	2500(0)	7500(0)	5000(0)
C(11)	1985(8)	-1983(7)	3911(13)
C(12)	1413(10)	-1380(11)	4792(15)
Atoms in the disordered tetraethylammonium group			
N(2)	2500(0)	2500(0)	5299(9)
C(13)[c]	2374(13)	1807(10)	4196(19)
C(13)*[c]	2374(13)	1807(10)	6402(19)
C(14)[c]	2247(9)	0972(7)	5299(15)

(a) Standard deviations of the least significant digits estimated by least squares are given in parentheses.

Table I (continued)

(b) The numbering system used for the anion is shown in Figure 1.

(c) C(13) and C(13)* represent disordered half atoms. Constraints forced on the parameters of the model of this disordered tetraethylammonium group are: x and y of C(13)* equal x and y of C(13), z of C(13)* was kept as much above the xy plane through N(2) as C(13) was below, and z of C(14) equals z of N(2).

Table II

Anisotropic Thermal Parameters[a]

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
			Atoms in the $Co(B_7C_2H_9)_2^-$ anion			
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±1.0	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±1.0	-0.8±.6	1.1±.8	-0.6±.7
B(8)	7.5±1.0	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±1.1	1.1±.7	0.2±.8	-0.2±.8
			Atoms in the ordered tetraethylammonium group			
N(1)[b]	6.8±.6	6.8±.6	3.9±.8	0.0±0	0.0±0	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±1.0	13.9±1.3	7.7±.9	5.3±.9	1.4±.7	0.1±.8
			Atoms in the disordered tetraethylammonium group			
N(2)[b]	5.2±.5	5.2±.5	4.6±.8	0.0±0	0.0±0	0.0±0
C(13)[c]	8.8±1.2	7.8±.9	7.0±.9	0.4±1.0	0.4±1.1	-4.0±.9
C(13)*[c]	8.8±1.2	7.8±.9	7.0±.9	0.4±1.0	-0.4±1.1	4.0±.9
C(14)	10.6±.9	3.6±.5	14.5±1.0	-0.5±.5	-0.7±.7	-2.0±.6

(a) The anisotropic thermal parameters, B, in units of $Å^2$, are given by $B_{ij} = 4\beta_{ij}/a_i^* a_j^*$, where a_i^* is the length of the ith reciprocal cell dimension.

Table II(continued)

(b) Symmetry conditions require $B_{11} = B_{22}$ and $B_{12} = B_{13} = B_{23} = 0$.

(c) Constraints forced on the disordered half atom in order to preserve sphenoidal symmetry are: B_{11}, B_{22}, B_{33} and B_{12} of C(13)* equal the corresponding values of C(13) and B_{13} and B_{23} of C(13)* are the negitive of those of C(13).

Table III

Observed and Calculated structure Factors. Each reflection given zero weight in the final refinement is indicated with an asterisk.

OBSERVED AND CALCULATED STRUCTURE FACTORS
FCA(0,0,0) = 2112

H	FOB	FCA
K,L= 0, 0		
2	273	311
4	149	182
6	22	40
8	66	78
10	243	245
12	235	216
14	67	61
16	61	56
K,L= 1, 0		
1	26	30
3	303	304
5	64	61
7	84	71
9	14	16
11	66	64
13	0	10*
15	0	22*
K,L= 2, 0		
2	444	454
4	172	172
6	116	141
8	102	111
10	176	156
12	144	132
14	87	87
16	63	61
K,L= 3, 0		
1	265	259
3	40	46
5	148	124
7	20	35
9	57	56
11	79	70
13	0	1*
15	0	6*
K,L= 4, 0		
2	14	23
4	298	301
6	235	222
8	199	197
10	178	165
12	58	51
14	19	21
16	71	72
K,L= 5, 0		
1	170	158
3	88	76
5	107	90
7	110	103
9	0	5*
11	0	2*
13	0	4*
15	0	9*
K,L= 6, 0		
2	191	227
4	282	228
6	297	298
8	267	267
10	211	199
12	81	76
14	109	106
16	55	55
K,L= 7, 0		
1	47	58
3	89	88
5	34	40
7	39	51
9	0	8*
11	105	105
13	24	27
15	19	14
K,L= 8, 0		
2	88	70
4	213	180
6	197	191
8	156	158
10	117	105
12	102	92
14	77	76
K,L= 9, 0		
1	27	24
3	15	15
5	16	20
7	0	21*
9	0	17*
11	36	38
13	14	7
K,L= 10, 0		
2	229	217
4	257	259
6	141	138
8	0	7*
10	87	82
12	59	62
14	75	84
K,L= 11, 0		
1	150	149
3	0	6*
5	0	28*
7	26	23
9	0	6*
11	27	16
K,L= 12, 0		
2	150	139
4	203	192
6	23	18
8	43	41
10	58	54
12	77	78
K,L= 13, 0		
1	39	35
3	0	7*
5	72	64
7	0	13*
9	14	19
K,L= 14, 0		
2	110	103
4	61	60
6	58	59
8	45	36
10	46	45
K,L= 15, 0		
1	14	17
3	27	17
5	14	1
7	19	18
K,L= 16, 0		
2	49	51
4	78	72
6	81	89
K,L= 0, 1		
0	492	599
1	23	23
2	786	817
3	14	13
4	124	123
5	14	18
6	153	147
7	23	25
8	31	31
9	23	24
10	215	216
11	47	29
12	178	176
13	32	12
14	96	97
15	14	2
16	52	55
K,L= 1, 1		
1	0	14*
2	28	28
3	218	218
4	20	20
5	0	13*
6	22	10
7	0	20*
8	9	13
9	0	3*
10	16	12
11	41	33
12	12	14
13	49	40
14	0	10*
15	14	11
K,L= 2, 1		
1	56	54
2	108	116
3	54	50
4	155	186
5	44	38
6	178	170
7	9	7*
8	123	109
9	45	46
10	223	209
11	35	20
12	149	140
13	26	10
14	54	53
15	0	2*
16	70	66
K,L= 3, 1		
1	260	248
2	36	38
3	151	140
4	33	38
5	82	70
6	66	60
7	13	40
8	0	8*
9	24	32
10	0	6*
11	48	53
12	0	6*
13	0	18*
14	0	4*
15	0	2*
K,L= 4, 1		
1	0	10*
2	162	180
3	49	46
4	210	212
5	43	40
6	322	311
7	21	16
8	228	215
9	11	16
10	125	109
11	0	8*
12	22	20
13	0	10*
14	72	79
15	0	8*
16	62	61
K,L= 5, 1		
1	157	120
2	37	41
3	8	12*
4	8	19*
5	27	29
6	38	36
7	42	39
8	15	16
9	0	15*
10	0	4*
11	12	28
12	0	9*
13	0	4*
14	0	7*
15	23	17
K,L= 6, 1		
1	25	17
2	62	79
3	12	11
4	309	293
5	29	29
6	237	219
7	37	43
8	263	279
9	16	18
10	176	167
11	0	7*
12	95	91
13	0	7*
14	70	61
16	55	59
K,L= 7, 1		
1	71	71
2	0	5*
3	32	30
4	60	50
5	0	5*
6	0	5*
7	15	16
8	0	3*
9	38	42
10	13	15
11	35	27
12	0	6*
13	33	27
14	14	16
K,L= 8, 1		
1	0	8*
2	134	139
3	34	28
4	243	217
5	0	6*
6	137	146
7	0	9*
8	116	116
9	28	22
10	142	127
11	0	13*
12	79	78
13	0	10*
14	88	91
15	18	11
K,L= 9, 1		
1	14	22
2	0	3*
3	11	6
4	26	27
5	25	26
6	0	3*
7	27	28
8	12	24
9	52	51
10	0	17*
11	0	3*
12	0	0*
K,L= 10, 1		
1	0	7*
2	180	184
3	30	35
4	211	200
5	20	27
6	121	110
7	0	2*
8	84	80
9	0	12*
10	51	49
11	0	0*
12	74	78
14	54	61
K,L= 11, 1		
1	62	61
2	17	13
3	68	64
4	17	25
5	25	28
6	0	2*
7	13	8
8	13	16
9	0	5*
10	0	16*
11	14	11
K,L= 12, 1		
1	0	1*
2	210	187
3	13	11
4	117	110
5	23	24
6	81	88
7	19	17
8	0	7*
9	0	2*
10	85	82
12	62	70
K,L= 13, 1		
1	41	44
2	0	6*
3	54	51
4	23	17
5	33	43
6	0	3*
7	0	9*
8	0	1*
K,L= 14, 1		
1	19	18
2	59	61
3	14	12
4	112	107
5	0	0*
6	43	45
7	0	5*
8	50	48
10	48	50
K,L= 15, 1		
1	19	19
2	0	0*
3	14	15
4	0	4*
K,L= 16, 1		
2	71	66
4	59	60
6	69	76
K,L= 0, 2		
0	769	860
1	52	57
2	144	181
3	7	21*
4	99	132
5	11	13
6	110	96
7	13	11
8	153	157
9	11	25
10	151	146
11	30	45
12	179	176
13	13	3
14	80	82
15	0	0*
16	55	60
K,L= 1, 2		
1	14	18
2	65	64
3	27	32
4	26	26
5	135	119
6	45	48
7	0	0*
8	0	5*
9	72	80
10	11	17
11	12	16
12	0	10*
13	13	1
14	0	12*
15	0	1*
K,L= 2, 2		
1	54	55
2	247	277
3	73	75
4	200	250
5	28	26
6	315	255
7	9	10
8	150	153
9	26	29
10	153	145
11	17	27
12	112	107
13	0	11*
14	91	97
15	14	11
16	65	64
17	18	19
K,L= 3, 2		
1	27	33
2	67	70
3	79	54
4	39	39
5	62	61
6	0	1*
7	25	25
8	31	36
9	22	25
10	0	9*
11	0	17*
12	0	7*
13	0	6*
14	0	3*
15	19	11
K,L= 4, 2		
1	24	22
2	168	170
3	86	69
4	315	318
5	33	31
6	228	221
7	10	16
8	248	239
9	32	31
10	114	102
11	18	20
12	45	57
13	0	17*
14	41	36
16	69	68
K,L= 5, 2		
1	24	19
2	72	56
3	39	43
4	82	62
5	69	61
6	37	37
7	23	27
8	33	38
9	38	41
10	0	12*
11	0	12*
12	0	2*
13	0	13*
14	0	12*
K,L= 6, 2		
1	24	20
2	265	250
3	25	18
4	137	145
5	35	33
6	234	226
7	0	10*
8	180	178
9	17	26
10	135	125
11	13	21
12	42	41
13	0	12*
14	89	79
16	45	46
K,L= 7, 2		
1	80	62
2	36	34
3	14	21
4	0	8*
5	10	1
6	37	42
7	16	33
8	0	17*
9	28	27
10	0	1*
11	46	38
12	27	22
13	0	9*
K,L= 8, 2		
1	22	28
2	76	81
3	0	5*
4	150	141
5	54	63
6	129	141
7	17	15
8	157	154
9	22	26
10	99	86
11	0	8*
12	87	79
13	0	2*
14	62	64
K,L= 9, 2		
1	11	6
2	0	9*
3	11	15
4	36	44
5	12	21
6	0	3*
7	12	10
8	31	35
9	13	13
10	0	13*
11	30	23
12	14	4
K,L= 10, 2		
1	0	4*
2	170	155
3	26	33
4	154	155
5	32	36
6	114	104
7	18	19
8	49	41
9	0	3*
10	94	92
11	0	7*
12	68	73
14	60	72
K,L= 11, 2		
1	34	34
2	12	2
3	12	5
4	31	33
5	0	7*
6	0	2*
7	13	10
8	19	18
9	0	1*
10	14	17
K,L= 12, 2		
1	18	19
2	117	109
3	43	47
4	138	133
5	0	1*
6	40	37
7	0	2*
8	71	67
9	14	12
10	75	73
12	68	79
K,L= 13, 2		
1	27	18
2	0	0*
3	0	10*
4	13	11
5	0	12*
6	0	7*
7	0	8*
8	0	0*
K,L= 14, 2		
1	14	7
2	108	108
3	0	2*
4	75	69
5	19	13
6	14	73
8	56	61
10	51	56
K,L= 15, 2		
1	19	8
2	0	9*
3	14	3
K,L= 16, 2		
2	61	59
4	57	53
6	58	69
K,L= 0, 3		
0	119	101
1	29	30
2	149	187
3	18	19
4	166	174
5	22	19
6	208	195
7	45	50
8	58	68
9	11	7
10	145	144
11	13	26
12	122	112
13	0	17*
14	96	100
16	52	63
K,L= 1, 3		
1	31	25
2	25	24
3	85	74
4	0	9*
5	90	85
6	19	15
7	34	34
8	19	19
9	43	44
10	0	8*
11	76	82
12	13	13
13	14	10
14	0	9*
K,L= 2, 3		
1	52	44
2	229	213
3	55	49
4	207	204
5	29	33
6	211	205
7	0	5*
8	226	226
9	20	21
10	112	100
11	13	21
12	115	117
13	14	9
14	74	69
16	50	56
K,L= 3, 3		
1	11	5
2	8	23*
3	78	70
4	27	25
5	57	59
6	41	41
7	21	23
8	16	19
9	58	53
10	0	3*
11	51	46
12	13	1
13	0	4*
14	14	6
K,L= 4, 3		
1	26	14
2	223	227
3	0	22*
4	182	203
5	73	62
6	321	297
7	0	8*
8	170	165
9	17	18
10	89	86
11	0	3*
12	30	30
13	0	15*
14	86	80
16	52	57
K,L= 5, 3		
1	118	107
2	13	22
3	0	7*
4	60	58
5	17	14
6	80	80
7	50	66
8	0	10*
9	0	2*
10	13	20
11	0	4*
12	0	7*
13	0	9*
K,L= 6, 3		
1	10	3
2	119	132
3	17	16
4	220	192
5	15	25
6	89	89
7	11	1
8	177	173
9	18	22
10	58	52
11	0	6*
12	56	52
13	14	17
14	52	43
K,L= 7, 3		
1	25	25
2	43	41
3	0	6*
4	11	1
5	0	19*
6	23	29
7	24	21
8	18	10
9	44	57
10	0	7*
11	0	7*
12	0	3*
13	0	10*
K,L= 8, 3		
1	11	17
2	189	187
3	16	13
4	110	115
5	26	23
6	119	114
7	18	23
8	113	105
9	0	6*
10	105	97
11	0	10*
12	14	66
14	55	60
K,L= 9, 3		
1	48	54
2	11	11
3	37	36
4	21	23
5	40	46
6	12	7
7	0	2*
8	32	30
9	27	24
10	0	13*
11	0	15*
K,L= 10, 3		
1	12	15
2	95	83
3	0	1*
4	101	90
5	22	17
6	93	81
7	0	11*
8	105	95
9	14	12
10	82	75
12	70	74
K,L= 11, 3		
1	0	11*
2	18	19
3	0	9*
4	26	27
5	19	8
6	0	12*
7	0	6*
8	0	6*
9	14	14
K,L= 12, 3		
1	0	9*
2	127	120
3	19	13
4	68	64
5	30	26
6	72	77
7	31	21
8	55	52
10	79	83
K,L= 13, 3		
1	0	12*
2	0	0*
3	14	6
4	19	12
5	0	2*
6	14	9
7	0	10*
9	19	28
K,L= 14, 3		
1	0	4*
2	82	80
3	19	17
4	79	83
6	66	70
7	19	4
8	66	65
K,L= 16, 3		
2	52	56
4	40	43
K,L= 0, 4		
0	123	121
1	30	27
2	150	148
3	21	11
4	109	104
5	25	16
6	100	109
7	11	15
8	178	181
9	30	34
10	65	57
11	0	14*
12	114	113
13	14	6
14	74	75
16	37	47
K,L= 1, 4		
1	30	26
2	9	5*
3	90	84
4	14	11
5	93	85
6	0	2*
7	30	37
8	0	4*
9	94	93
10	0	3*
11	37	35
12	0	3*
13	0	3*
15	19	17
K,L= 2, 4		
1	24	20
2	141	149
3	76	77
4	183	181
5	0	2*
6	225	220
7	25	32
8	108	115
9	0	5*
10	112	107
11	0	10*
12	81	75
13	0	16*
14	74	75
K,L= 3, 4		
1	23	28
2	30	27
3	24	17
4	18	17
5	15	21
6	25	29
7	11	12
8	0	6*
9	57	59
10	0	16*
11	58	55
12	0	7*
13	0	8*
K,L= 4, 4		
1	48	51
2	209	209
3	45	49
4	237	217
5	42	47
6	123	126
7	12	18
8	191	180
9	0	17*
10	52	49
11	0	13*
12	68	71
13	0	13*
14	52	53
K,L= 5, 4		
1	0	2*
2	41	44
3	39	43
4	0	15*
5	22	18
6	0	8*
7	0	21*
8	58	64
9	46	44
10	0	6*
11	0	8*
12	0	9*
K,L= 6, 4		
1	61	60
2	220	216
3	38	37
4	71	67
5	12	12
6	139	124
7	0	13*
8	88	75
9	0	20*
10	59	59
11	0	8*
12	41	38
14	55	53
K,L= 7, 4		
1	11	11
2	11	22
3	20	24
4	66	73
5	36	37
6	12	16
7	59	53
8	26	29
9	27	26
10	0	0*
11	14	21
12	0	3*
K,L= 8, 4		
1	45	44
2	94	93
3	29	37
4	128	118
5	45	36
6	102	97
7	45	53
8	129	117
9	0	7*
10	75	70
11	0	12*
12	55	59
13	18	14
K,L= 9, 4		
1	0	8*
2	12	7
3	18	17
4	0	9*
5	58	54
6	0	3*
7	13	23
8	0	11*
9	19	19
10	0	5*
K,L= 10, 4		
1	26	27
2	101	106
3	18	27
4	68	77
5	0	2*
6	68	76
7	0	8*
8	58	69
9	0	17*
10	76	76
12	57	65
K,L= 11, 4		
1	53	45
2	0	0*
3	13	14
4	13	15
5	0	6*
6	14	10
7	0	2*
8	0	2*
K,L= 12, 4		
1	14	6
2	80	71
3	19	22
4	65	67
5	0	3*
6	49	51
7	0	3*
8	63	63
10	54	64
K,L= 13, 4		
1	14	3
2	0	6*
3	0	7*
4	0	9*
K,L= 14, 4		
1	19	12
2	71	76
4	55	53
6	58	65
K,L= 16, 4		
1	21	22
K,L= 0, 5		
0	114	118
1	15	19
2	115	117
3	27	23
4	128	127
5	20	8
6	132	133
7	46	46
8	66	58
9	37	37
10	104	98
11	0	4*
12	68	71
14	52	59
K,L= 1, 5		
1	24	23
2	15	16
3	70	70
4	0	16*
5	33	33
6	17	26
7	0	1*
8	0	6*
9	0	14*
10	0	3*
11	47	42
12	14	1
13	19	17
K,L= 2, 5		
1	62	70
2	157	161
3	0	7*
4	120	113
5	16	20
6	90	89
7	17	15
8	144	136
9	26	24
10	55	51
11	0	4*
12	82	81
14	45	46
K,L= 3, 5		
1	19	35
2	0	2*
3	45	59
4	0	11*
5	12	13
6	0	11*
7	0	1*
8	22	24
9	40	44
10	0	0*
11	0	17*
12	14	7
13	19	10
K,L= 4, 5		
1	40	44
2	168	162
3	20	28
4	105	92
5	12	12
6	151	145
7	34	38
8	81	78
9	13	11
10	71	66
11	0	2*
12	53	58
14	57	60
K,L= 5, 5		
1	0	6*
2	20	18
3	17	21
4	43	44
5	35	13
6	44	39
7	41	32
8	0	4*
9	13	7
10	27	21
11	0	2*
K,L= 6, 5		
1	0	3*
2	107	106
3	0	10*
4	126	119
5	13	18
6	48	51
7	56	52
8	93	85
9	0	4*
10	43	44
11	0	16*
12	53	54
K,L= 7, 5		
1	17	13
2	25	30
3	0	1*
4	62	58
5	22	24
6	26	33
7	0	8*
8	14	5
9	31	31
10	0	3*
K,L= 8, 5		
1	13	8
2	154	149
3	56	50
4	92	89
5	30	24
6	98	93
7	36	35
8	83	79
9	33	34
10	68	68
12	48	51
K,L= 9, 5		
1	39	38
2	0	10*
3	44	41
4	13	1
5	52	48
6	14	5
7	14	14
8	19	14
K,L= 10, 5		
1	30	22
2	66	65
3	49	39
4	79	75
5	19	8
6	75	73
7	14	15
8	72	64
10	52	57
K,L= 11, 5		
1	0	6*
2	0	6*
3	0	1*
4	0	9*
5	14	6
6	14	11
K,L= 12, 5		
1	0	9*
2	70	71
3	14	11
4	45	46
6	52	54
8	31	38
K,L= 14, 5		
2	45	46
4	38	45
K,L= 0, 6		
0	146	144
1	12	7
2	124	117
3	12	18
4	95	88
5	22	14
6	67	63
7	26	12
8	99	91
9	24	23
10	45	48
12	58	64
K,L= 1, 6		
1	12	19
2	0	3*
3	21	25
4	0	15*
5	13	10
6	18	12
7	0	9*
8	0	3*
9	24	30
10	0	2*
K,L= 2, 6		
1	24	25
2	122	117
3	18	17
4	71	63
5	0	17*
6	84	84
7	30	38
8	51	50
9	0	5*
10	67	63
12	47	54
K,L= 3, 6		
1	28	32
2	0	5*
3	33	36
4	0	3*
5	0	20*
6	0	1*
7	0	10*
8	0	8*
9	14	21
10	14	8
K,L= 4, 6		
1	18	17
2	115	105
3	0	11*
4	89	96
5	52	51
6	48	51
7	27	26
8	77	75
9	14	9
10	47	51
12	58	61
K,L= 5, 6		
1	34	32
2	0	12*
3	29	35
4	29	32
5	13	8
6	0	1*
7	0	2*
8	24	22
9	0	1*
K,L= 6, 6		
1	39	42
2	119	113
3	39	42
4	59	57
5	23	23
6	73	75
7	0	1*
8	36	52
9	19	20
10	52	54
12	43	48
K,L= 7, 6		
1	19	16
2	13	17
3	0	11*
4	33	33
5	14	19
6	0	7*
7	0	2*
8	19	15
K,L= 8, 6		
1	13	23
2	73	66
3	0	11*
4	95	91
5	0	3*
6	75	75
7	30	33
8	67	65
10	43	49
K,L= 9, 6		
1	14	18
2	14	10
3	14	15
4	0	5*
5	27	25
6	0	5*
K,L= 10, 6		
1	0	16*
2	81	77
3	19	13
4	69	69
5	19	4
6	60	60
8	36	41
K,L= 12, 6		
2	42	47
4	47	46
6	30	35
K,L= 0, 7		
0	102	86
1	13	14
2	113	106
3	13	1
4	71	62
5	0	12*
6	61	59
7	24	18
8	45	37
10	49	51
K,L= 1, 7		
1	13	24
2	13	14
3	0	7*
4	0	6*
5	0	3*
6	0	4*
7	14	7
8	0	5*
K,L= 2, 7		
1	0	5*
2	79	69
3	0	14*
4	54	57
5	19	21
6	33	39
7	0	12*
8	58	51
10	41	43
K,L= 3, 7		
1	30	25
2	0	9*
3	27	33
4	0	4*
5	0	2*
6	14	7
7	0	1*
K,L= 4, 7		
1	0	23*
2	86	82
3	23	18
4	36	43
5	0	17*
6	56	61
7	19	15
8	43	46
10	44	46
K,L= 5, 7		
1	23	20
2	0	8*
3	0	6*
4	14	1
5	0	7*
6	0	5*
K,L= 6, 7		
1	0	12*
2	56	55
3	0	19*
4	64	67
5	14	21
6	49	48
8	47	50
K,L= 7, 7		
1	0	0*
2	14	7
3	0	1*
4	24	14
7	19	8
K,L= 8, 7		
1	14	6
2	68	61
4	54	58
6	51	56
8	35	42
K,L= 9, 7		
1	19	2
K,L= 10, 7		
2	39	46
4	51	53
K,L= 0, 8		
0	94	86
1	24	15
2	55	44
3	19	17
4	57	52
6	30	38
8	36	32
K,L= 1, 8		
1	0	10*
2	14	17
3	14	15
K,L= 2, 8		
1	14	4
2	62	64
3	14	7
4	23	32
6	42	41
8	36	38
K,L= 3, 8		
1	24	21
2	0	5*
K,L= 4, 8		
2	41	44
4	44	49
6	37	37
7	18	12
K,L= 6, 8		
2	37	43
4	37	38
6	38	44
K,L= 8, 8		
2	25	30
K,L= 0, 9		
0	25	21
2	40	44
K,L= 2, 9		
1	22	19
2	22	24

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Table IV

Root Mean Square Vibrational Amplitudes for the $Co(B_7C_2H_9)_2^-$ Anion

	r.m.s. displacement(Å)		
Atom	Min	Med	Max
Co(2)	0.21	0.22	0.26
C(1)	0.20	0.24	0.30
B(3)	0.22	0.25	0.34
B(4)	0.24	0.26	0.36
B(5)	0.21	0.24	0.34
BC(6)	0.25	0.29	0.29
B(7)	0.21	0.30	0.35
B(8)	0.24	0.32	0.33
BC(9)	0.20	0.24	0.27
B(10)	0.24	0.30	0.37

cage, the distances to the carbon are shorter than the distances to the corresponding boron. Thus, some of the atoms in the cage are shifted closer to position 6 in some of the cages and closer to position 9 in the other cages, depending on the position of the carbon. Since we observe an average effect in the X-ray analysis, it appears as though the atoms in the cage have large thermal motion when in reality, the temperature factors are attempting to accommodate two atoms displaced slightly from each other. It is more likely that the thermal parameters are describing disorder rather than thermal motion since, as is well displayed by the stereoscopic pair drawing shown in Figure 2, no clear pattern of collective motion is shown by the thermal ellipsoids (e.g. bending motion of the cages across the cobalt, etc.). Also, if a carbon is disordered between positions 6 and 9, it would be expected that B(10) would be affected more than any other atom in the cage, with its largest movement almost along the z direction. The component of the thermal ellipsoid for B(10) is the largest in the z direction and is larger than any component for any other atom in the cage. Thus, it appears likely that one carbon is localized at position 1 and the other carbon is disordered between positions 6 and 9. This disorder makes impossible a decision about the isomerism of any particular anion in the crystal. Let us say that a $Co(B_7C_2H_9)$ cage is a D cage if the carbons are in positions 1 and 6 and its mirror image in which the carbons occupy positions 1 and 9 is an L cage. Then for the $Co(B_7C_2H_9)_2^-$ anion, there are two possible synisomers, a D-D and an L-L, and only one antiisomer, a D-L. There are, however, two rotational orientations possible for the antiisomer in the crystal, a D-L and an L-D. The disorder found in this work may be accounted for in the following ways:

(1) all the $Co(B_7C_2H_9)_2^-$ anions are synisomers, half D-D and half L-L, (2) all the anions are antiisomers and the probability that an anion will pack with a D-L orientation is equal to the probability that it will pack with an L-D orientation, or (3) any combination of the first two.

The disordered tetraethylammonium group may be pictured as two interpenetrating sphenoidal tetraethylammonium groups with their $\bar{4}$ axes coincident. One group is rotated 90° about the $\bar{4}$ axis from the position in which all atoms are coincident, thus giving the entire group 4-fold rotational symmetry. This 4-fold axis is along the z direction of the unit cell. The two carbon half atoms in this disordered tetraethylammonium group, C(13) and C(13)*, are separated by 1.89 Å in the z direction.

Interatomic distances for all atoms are given in Table V and interatomic angles in the anion are given in Table VI. The standard deviations quoted are very optimistic estimates since they are calculated from least squares estimates of the accuracy of the atomic positions and do not explicitly take into account the effects of the disorder. Because of this disorder in the cages, distances and angles involving BC(6) and BC(9) have little chemical meaning since both of these positions are averages of the values arising when the position is occupied by a carbon or a boron. As expected, the distances to atoms at the apices, C(1) and B(10), are shorter than distances to the non-apical atoms because of their lower coordination. The longer distances to B(5) and B(3) from the cobalt are a result of the non-planarity of the open faces of the polyhedral fragments.

The molecular packing is shown in Figure 3 by a projection of the

Table V

Interatomic Distances[a]

Atom 1	Atom 2	Distance(Å)
Atoms in the $Co(B_7C_2H_9)_2^-$ anion		
Co(2)	C(1)	1.93
Co(2)	BC(9)	2.05
Co(2)	BC(6)	2.05
Co(2)	B(3)	2.15
Co(2)	B(5)	2.15
C(1)	B(3)	1.57
C(1)	B(5)	1.60
C(1)	B(4)	1.64
B(3)	BC(6)	1.79
B(3)	B(4)	1.87
B(3)	B(7)	1.90
B(4)	B(8)	1.79
B(4)	B(7)	1.79
B(4)	B(5)	1.89
B(5)	BC(9)	1.76
B(5)	B(8)	1.84
BC(6)	BC(9)	1.73
BC(6)	B(7)	1.82
B(7)	B(8)	1.89
B(8)	BC(9)	1.82
B(10)	BC(6)	1.68
B(10)	BC(9)	1.68
B(10)	B(7)	1.71

Table V(continued)

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 from least squares estimates of the accuracy of the coordinates are ±0.01 Å for distances to Co(2) and ±0.02 Å for all other distances.

Table VI

Interatomic Angles in the $Co(B_7C_2H_9)_2^-$ Anion

Atom 1	Atom 2	Atom 3	Angle(degrees)	Atom 1	Atom 2	Atom 3	Angle(degrees)
Angles around 3-membered rings							
C(1)	Co(2)	B(3)	44.9 ± 0.5	C(1)	Co(2)	B(5)	45.7 ± 0.5
B(3)	Co(2)	BC(6)	50.3 ± 0.5	B(5)	Co(2)	BC(9)	49.3 ± 0.5
BC(6)	Co(2)	BC(9)	50.0 ± 0.5	B(3)	C(1)	B(4)	71.3 ± 0.8
B(4)	C(1)	B(5)	71.4 ± 0.8	C(1)	B(3)	B(4)	56.1 ± 0.7
C(1)	B(4)	B(3)	52.6 ± 0.7	C(1)	B(4)	B(5)	53.4 ± 0.7
C(1)	B(5)	B(4)	55.3 ± 0.7	B(3)	B(4)	B(7)	62.3 ± 0.8
B(4)	B(3)	B(7)	56.9 ± 0.7	B(3)	B(7)	B(4)	60.7 ± 0.8
B(3)	B(7)	BC(6)	57.4 ± 0.7	B(3)	BC(6)	B(7)	63.6 ± 0.7
BC(6)	B(3)	B(7)	59.0 ± 0.7	BC(6)	B(7)	B(10)	56.7 ± 0.8
B(7)	BC(6)	B(10)	58.3 ± 0.8	BC(6)	B(10)	B(7)	65.0 ± 0.8
BC(6)	BC(9)	B(10)	58.9 ± 0.8	BC(9)	BC(6)	B(10)	58.9 ± 0.8
BC(6)	B(10)	BC(9)	62.2 ± 0.8	B(8)	B(10)	BC(9)	63.8 ± 0.8
B(8)	BC(9)	B(10)	60.3 ± 0.8	BC(9)	B(8)	B(10)	55.9 ± 0.8
B(5)	B(8)	BC(9)	57.4 ± 0.7	B(5)	BC(9)	B(8)	61.8 ± 0.7
B(8)	B(5)	BC(9)	60.7 ± 0.7	B(5)	B(4)	B(8)	59.8 ± 0.8
B(4)	B(5)	B(8)	57.6 ± 0.8	B(4)	B(8)	B(5)	62.6 ± 0.8
B(4)	B(7)	B(8)	58.3 ± 0.8	B(7)	B(4)	B(8)	63.4 ± 0.8
B(4)	B(8)	B(7)	58.4 ± 0.8	B(7)	B(8)	B(10)	55.7 ± 0.8
B(8)	B(7)	B(10)	58.4 ± 0.9	B(7)	B(10)	B(8)	65.9 ± 0.9
Angles around 4-membered rings							
Co(2)	B(3)	B(4)	100.0 ± 0.8	Co(2)	B(5)	B(4)	99.1 ± 0.8
B(3)	Co(2)	B(5)	73.9 ± 0.5	B(3)	B(4)	B(5)	87.0 ± 0.8

Table VI (continued)

BC(6)	B(7)	B(8)	88.4 ± 0.9	B(7)	B(8)	BC(9)	86.7 ± 0.8
BC(6)	BC(9)	B(8)	93.3 ± 0.8	B(7)	BC(6)	BC(9)	91.5 ± 0.8
Angles around 5-membered rings							
C(1)	B(3)	BC(6)	108.0 ± 1.0	C(1)	B(5)	BC(9)	107.9 ± 0.9
B(3)	BC(6)	BC(9)	103.5 ± 0.9	B(5)	BC(9)	BC(6)	104.4 ± 0.8
B(3)	C(1)	B(5)	109.5 ± 0.9	C(1)	Co(2)	BC(6)	86.1 ± 0.5
C(1)	B(4)	B(7)	109.5 ± 0.9	Co(2)	BC(6)	B(7)	117.9 ± 0.8
B(4)	B(7)	BC(6)	99.7 ± 0.9	Co(2)	C(1)	B(4)	119.3 ± 0.7
C(1)	B(3)	B(7)	107.8 ± 1.0	C(1)	B(5)	B(8)	108.3 ± 1.0
B(3)	B(7)	B(8)	100.2 ± 0.9	B(5)	B(8)	B(7)	101.4 ± 0.9
C(1)	Co(2)	BC(9)	85.9 ± 0.4	C(1)	B(4)	B(8)	108.6 ± 1.0
B(4)	B(8)	BC(9)	99.7 ± 0.9	Co(2)	BC(9)	B(8)	117.7 ± 0.7
Co(2)	B(3)	B(7)	110.0 ± 0.8	Co(2)	BC(9)	B(10)	123.3 ± 0.8
B(3)	Co(2)	BC(9)	82.3 ± 0.5	B(3)	B(7)	B(10)	110.3 ± 1.0
B(7)	B(10)	BC(9)	97.5 ± 1.0	B(3)	B(4)	B(8)	104.8 ± 0.9
B(3)	BC(6)	B(10)	117.4 ± 0.9	B(4)	B(3)	BC(6)	98.1 ± 0.9
BC(6)	B(10)	B(8)	97.4 ± 1.0	B(4)	B(8)	B(10)	109.5 ± 1.1
B(4)	B(5)	BC(9)	98.4 ± 0.9	B(4)	B(7)	B(10)	111.9 ± 1.1
B(5)	B(4)	B(7)	102.8 ± 0.9	B(5)	BC(9)	B(10)	117.6 ± 0.9
Co(2)	B(5)	B(8)	112.0 ± 0.8	Co(2)	BC(6)	B(10)	123.3 ± 0.9
B(5)	Co(2)	BC(6)	81.9 ± 0.5	B(5)	B(8)	B(10)	109.5 ± 1.0

unit cell onto the xy plane. Only half of the unit cell is shown (x, 0 to 1; y, 0 to $\frac{1}{2}$) since the other half is related to this by a center of symmetry at $\frac{1}{2},\frac{1}{2},0$. The disordered tetraethylammonium group with N(2) lying on a 4-fold axis is shown with its bonds dashed to distinguish it from the tetrahedral group around N(1) shown in solid lines. The cages are lying nearly in the xy plane with z = 0 and z = 1 for all the cobalt atoms. The tetraethylammonium groups are situated with z = $\frac{1}{2}$ for N(1) and z = 0.53 for N(2), displaced 0.26 Å higher along the z direction than N(1).

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Figure Captions

Figure 1: Skeletal drawing of the $Co(B_7C_2H_9)_2^-$ anion showing the structure, the positions of the carbon atoms, and the numbering system used.

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

Figure 3: View of half of the unit cell ($\underline{x}$, 0 to 1; $\underline{y}$, 0 to $\frac{1}{2}$) looking down the $\underline{c}$ axis of the unit cell. The other half of the cell is related to this by a center of symmetry at $\frac{1}{2},\frac{1}{2},0$. The cobalt atoms are at $\underline{z} = 0$ and the nitrogen atoms are near $\underline{z} = \frac{1}{2}$. The ordered tetraethylammonium group is shown with solid bonds and the disordered group is shown with dashed bonds.

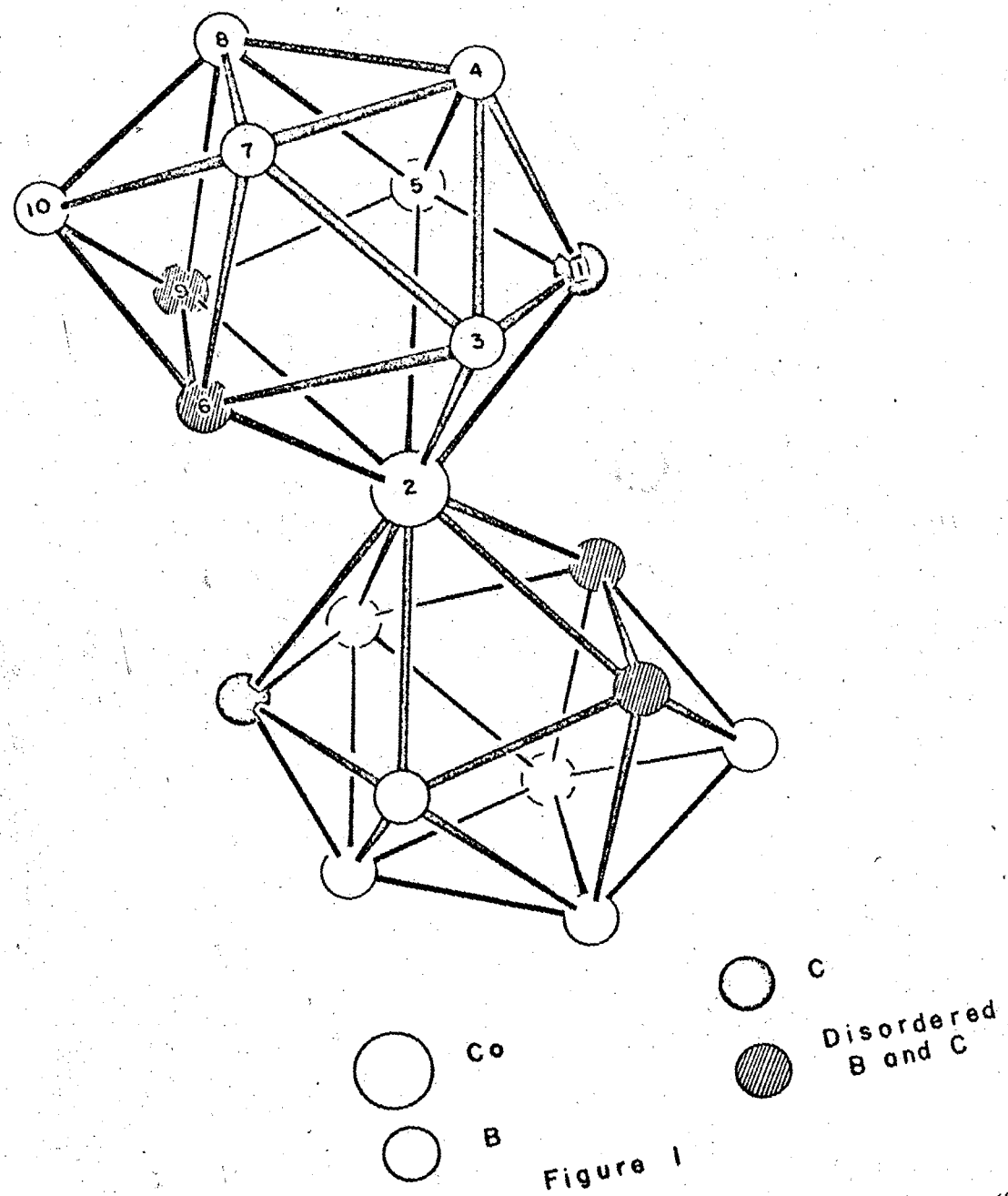

Figure 1

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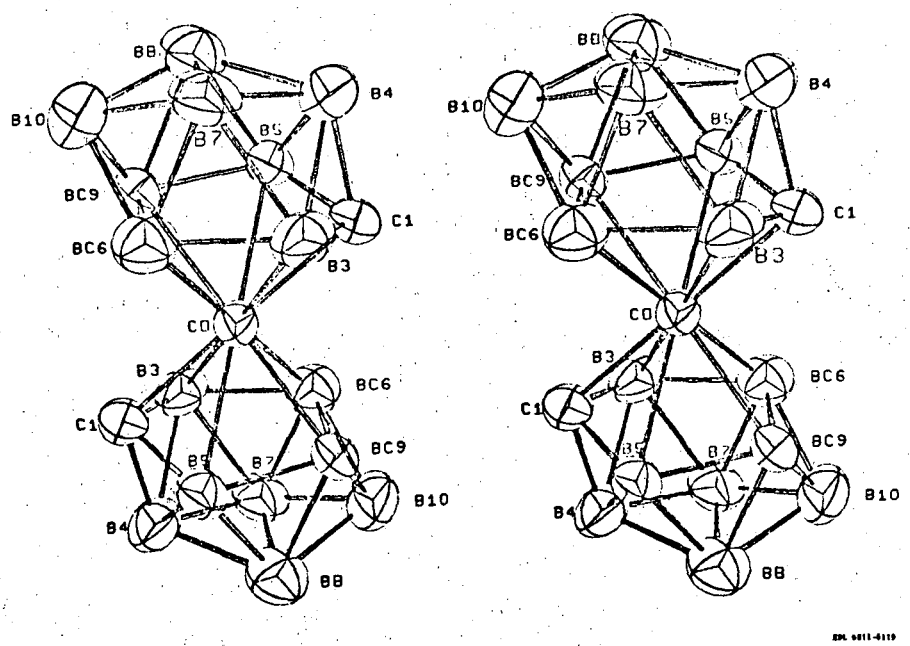
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B4
B10
B7
B5
BC9
C1
BC6
B3
CO
B3
BC6
C1
BC9
B5
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B10
B4
B8
B8
B4
B10
B7
B5
BC9
C1
BC6
B3
CO
B3
BC6
C1
BC9
B5
B7
B10
B4
B8

Fig. 2

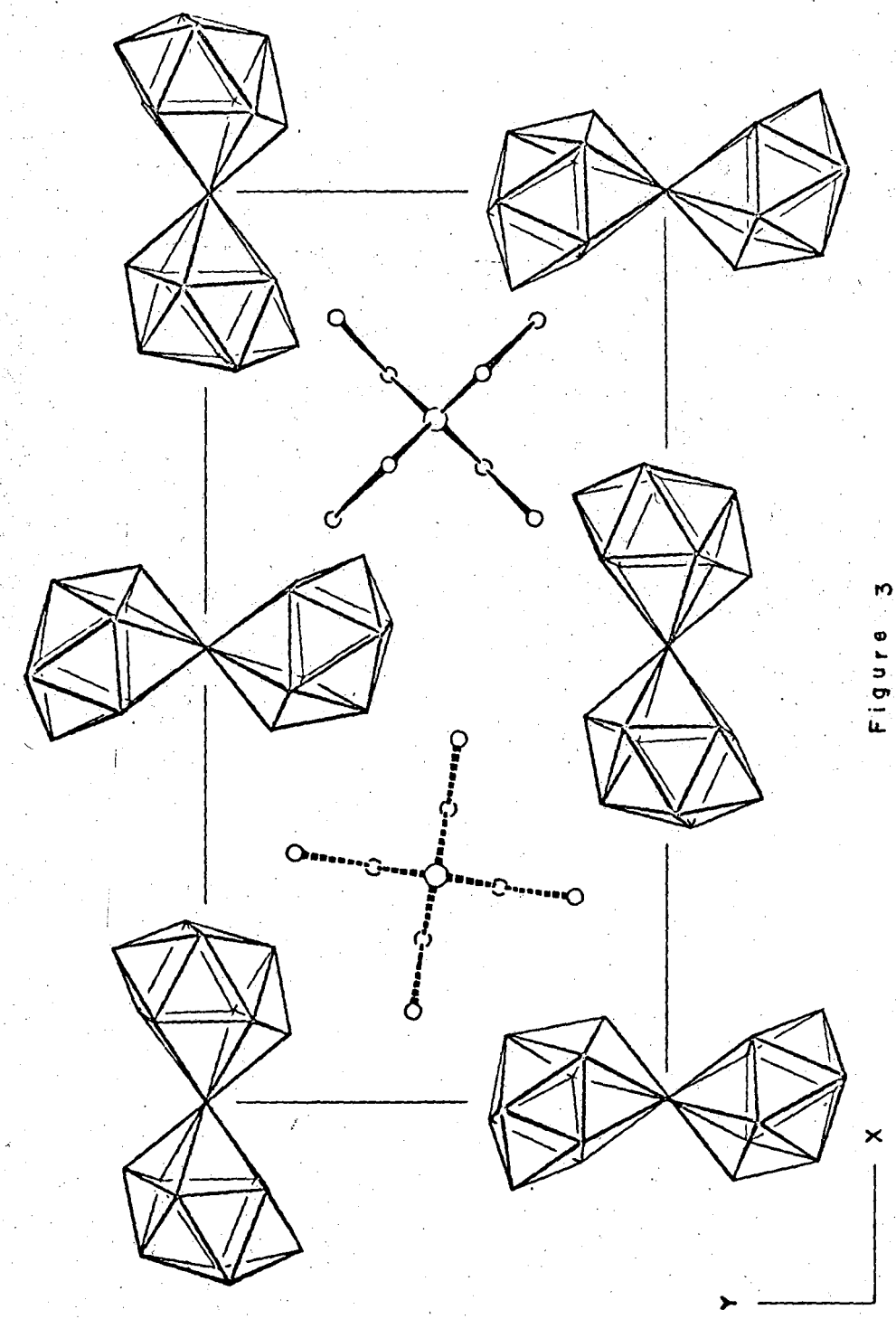

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Figure 3

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